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The Deacidification of Cellulosic Textiles

by

Teena (Rentenaar) Jennings

(C)

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE

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Supervisor

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Date.....Nov. 21, 1984

ABSTRACT

The Deacidification of Cellulosic Textiles

by

Teena (Rentenaar) Jennings, Master of Science

University of Alberta, 1985

Professor: Dr. Nancy Kerr

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Researchers have shown that deacidification agents are effective in slowing down the degradation of cellulosic products. Deacidificants are currently being used by paper conservators, however, textile conservators remain hesitant in adopting these agents for routine treatment. There are concerns about the possible damaging effects which deacidificants may have on textile fibres. Any permanent damage caused by the presence of the deacidifying agents would negate the possibility of removing the treatment without residual effects. There are also concerns regarding a colour change of the textile upon treatment with deacidifying agents. Additionally, conservators wonder about an increased stiffness which might alter the aesthetic appeal of the textile. Finally, the hygroscopic nature of the magnesium salts has been cited as a problem since there may be an increased propensity for the agents to deliquesce.

A variety of textiles were used in this study to address these concerns. They included new, undyed cotton fabric; naturally aged cellulosic textiles; cotton fabric aged by γ-irradiation and new, dyed, commercially available cotton.

Three deacidification agents were used: aqueous-based magnesium bicarbonate, nonaqueous Wei T'o® solution #2, a formulation for dipping specimens and nonaqueous Wei T'o® solution #12 spray.

The results of this study show that there is only minimal colour change of textiles upon treatment with deacidification agents. Textiles which contain many yellow water-soluble degradation products should be washed before treatment if possible as was indicated by studies carried out on γ-irradiated cotton. Also, change in moisture regain for textiles which are deacidified can not be regarded as a problem based on the results herein as there was only a slight increase (0.2 - 0.4 %) over the moisture regain of the control fabrics. However, there was undoubtedly an increase in stiffness, especially with the application of the nonaqueous Wei T'o® solution #2. Dipping fabrics in Wei T'o® caused about a 10 fold increase in stiffness. Notwithstanding, this stiffness was lessened with only a minimal amount of handling. Finally, there was a reduction in abrasion resistance in the presence of the deacidification agents when testing new cotton, however, this was not the case when testing naturally aged cellulosic textiles.

The scanning electron micrographs show very few crystals; rather, there is an allover smooth coating. The energy dispersive X-ray analyses of the surface of the fibres indicate that magnesium is present eventhough crystals are not evident. These results indicate that damage to the fibres, as was presumed to be the case due to the presence of the crystals, need not be so.

This study should alleviate some of the concerns which textile conservators have in accepting deacidification agents as a treatment for historic textiles.

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At this time I would like to express my sincerest gratitude to those who helped to make the completion of this thesis a reality.

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1. INTRODUCTION

Curators and conservators of textiles are charged with the safekeeping of the textiles in their care. This safekeeping goes beyond the restorative work usually associated with conservators and includes methods which will slow down the ageing process, hence prolonging the life of historic textiles.

All organic materials, including textiles, undergo degradation, through the natural process of ageing. Conservators are aware of preventive measures which help to slow down the process, such as controlling the relative humidity and temperature of the environment and eliminating high energy light sources. This research involves the possibility of going one step further in controlling degradation, by applying an agent to the textile which will extend its life.

Textile conservators in general are slow to adopt new techniques of treatment, regardless of how theoretically sound a particular treatment may be. This is justifiable to a certain extent as historic artefacts are often irreplaceable. Conservators must think of the possible long term benefits of a treatment but alternatively must remain cautious. Regret does little to replace a valuable artefact.

It has been shown quite clearly that levels of acidity in cellulosic textiles such as cotton, linen and rayon, promote the degradation of these fibres. Moreover, acidity is a direct consequence of ageing. It has also been shown that the deacidification of cotton fabric slows down the degradative process (Kerr, 1982).

This study will focus on the physical changes which occur when cotton fabrics are deacidified in an attempt to assure conservators that such a treatment is appropriate. Evidence clearly points to the urgency of accepting treatments of this kind as cellulosic textiles continue to deteriorate in spite of careful storage and display. The current method of treatment, which is washing with deionized or distilled water, may reduce the acidity within the textile but does nothing to neutralize acids which form at a later date. In fact, evidence now suggests that using deionized or distilled water rather than tap water when washing paper actually leaves the

cellulose in a more reactive state by protonating the carboxyl groups and thus more prone to develop higher acidity levels (Tang and Jones, 1979). Kerr (1982) found that the treatment with distilled water offered the cotton textile no protection in further ageing.

Paper conservators are using alkaline treatments routinely (Waters, 1981). There are many reasons for paper conservators to more readily accept these treatments than textile conservators. Paper cellulose, due to its source and production, has more impurities than cotton cellulose. These impurities contribute to an increased rate of deterioration. Also, paper artefacts have traditionally had more monetary or historic value attributed to them than textiles, and therefore conservators were initially more anxious to begin using deacidification treatments. In addition, effects of the deacidification agent on the artefact can be more obtrusive on textiles than paper and this remains a concern for textile conservators.

1.1 Statement of the Problem

Studies have shown that cellulosic artefacts stored under museum conditions have high acidity levels (Dawson, 1980; Peacock, 1980). There are several sources of acidity. It may be produced within the cellulose molecule as the fabric ages. The acidity is evidence of oxidation which, in turn can be harmful to cellulosic textiles because an oxidized cellulose molecule shows an increased tendency to hydrolyze (Rånby, 1959).

Environmental pollutants, such as sulphur dioxide gas and oil-coated particulate matter, are inherently acidic or may cause acidic products to be formed. Treatments to cotton during and after its manufacture may leave residual acid present in the textile. Microorganisms, for example fungi and bacteria, produce enzymes which are acidic (Howard and McCord, 1959; Peacock, 1980). It is important to note that the detrimental effects of acids on cellulosic textiles is not dependent on the source or kind of acid.

In prolonging the life of a cellulosic textile, it is important to retard oxidative and hydrolytic reactions as much as possible because both of these reactions are harmful to cellulosic textiles. Hydrolysis causes the cellulose chain to rupture and thus reduces strength.

Oxidation causes the cellulose to yellow and imparts to the cellulose chain an increased propensity to hydrolyze. In addition, as acidic degradation products accumulate, the pH of the aqueous extract decreases.

Deacidification is a means of controlling acidity and is a treatment well accepted in the field of paper conservation (Hey, 1979). There is now evidence (Block, 1981; Kerr, 1982: Peacock, 1980) that this process is also beneficial to textiles in combatting the acidity levels. A definite obstacle to the use of deacidifying agents, however, is the unwillingness of textile conservators to use these agents on cellulosic textiles. There are numerous questions concerning their visual effect and the long term effect of the alkaline agents on the cellulosic substrate. The visual aspects are not as pressing in paper artefacts as in textiles because paper need not have the qualities of drapeability and softness that are so essential in the presentation of textiles. There is evidence that oxidized cellulose is apt to be degraded by strong bases (Peters, 1963). Will the alkalinity of deacidifying agents, therefore, damage the old cellulosic textiles which are oxidized with age and cause increased yellowing? How would these buffers aid or affect textiles which have aged naturally? Conservational treatments of textiles are usually avoided if they are not entirely reversible. Can a deacidification treatment be removed with no residual effects? These and other questions related to textile deacidification will be dealt with in this study. Therefore, it is the intent of this study to investigate the problems and questions which textile conservators have put forward concerning deacidifying agents on cellulosic textiles.

1.2 Justification

This study should be of interest to curators and conservators responsible for the care of cellulosic textiles, including such fibres as cotton, linen and rayon. Art conservators should also be interested as many paintings are done on linen or cotton canvas. Unpainted canvas, used as an integral design element of paintings is an interesting problem in that as uncontrolled ageing progresses, the colour of the canvas will change thus altering the visual effect of the painting. Therefore, keepers of paintings will also be interested in slowing degradation.

The washing of textiles to remove the acidic degradation products is not totally effective, as last traces of acid are very difficult to remove (Ant-Wuorinen and Visapää, 1963; Dawson, 1980). Residual acids left in the textile will cause further degradation (Peters, 1963). Also, the washing process does little to combat future acids that come into contact with the textile from environmental sources. Therefore, a treatment which will handle these situations would be important in fighting the imminent degradation.

There is now evidence of reduced degradation through the application of a deacidifying agent (Block,1981; Kerr, 1982; Peacock, 1980). In fact, it has been shown that treatment with some deacidificants actually reduces the rate of oxidation of cotton (Kerr, 1982).

This study is a direct consequence of work done by Kerr (1982). In the initial study by Kerr, it was shown that beneficial effects resulted from treating cotton with three alkaline buffers, calcium hydroxide, magnesium bicarbonate and methoxy magnesium methyl carbonate (known commercially as Wei T'o®). All three buffers provided protection against degradation under conditions of accelerated ageing with moist heat. The two most effective agents, magnesium bicarbonate and methoxy magnesium methyl carbonate, will be the focus of this study.

As the theoretical groundwork for deacidification has been established, there is a need to satisfy the current questions posed by conservators concerning the routine deacidification of cellulosic textiles. This process must be found to be acceptable, practical and workable from the conservator's point of view, or else this potentially valuable tool for extending the life of cellulosic textiles may never be used.

1.3 Objectives

The objectives of this study are:

a. - to determine if the application of the deacidifying agent affects the colour of new white cotton, white cotton which has been naturally aged and new white cotton which has been aged by accelerated means.

b. - to determine if the application of the deacidifying agent changes the colour of selected dyed textiles.

c. - to determine if the deacidifying agents adversely affect hand and stiffness.

d. - to determine the change in moisture regain of the deacidified cotton fabrics and thus the possibility of deacidifying agents to deliquesce.

e. - to determine if the deacidificants abrade the fibres of the textiles and thus negate the possibility of reversing the treatment.

f. - to determine the presence and location of the buffering particles within the fibres and yarns of the textiles.

g. - to determine the most effective means of applying the nonaqueous deacidifying agent, methoxy magnesium methyl carbonate, whether by dipping or spraying.

1.4 Null Hypotheses

The null hypotheses are:

a. There is no significant difference in the colour change of new white cotton textiles after treatment with each of the deacidifying agents, methoxy magnesium methyl carbonate (MMMC) dip, MMMC spray and $Mg(HCO_3)_2$ dip.

b. There is no significant difference in the colour change of naturally aged white cellulosic textiles after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

c. There is no significant difference in the colour change of white cotton textiles aged by accelerated means after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

d. There is no significant difference in the colour change of selected commercially dyed cotton textiles after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$dip.

e. There is no significant difference in the stiffness of warp or weft specimens of new

cotton textiles after treatment with distilled water and the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

f. There is no significant difference in the moisture regain of new cotton textiles treated with distilled water and the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

g. There is no significant difference in the number of cycles required to rupture new cotton textiles, through flexing and abrading, among the four treatments, distilled water, MMMC dip, MMMC Spray and $Mg(HCO_3)_2$ dip.

h. There is no significant difference in the number of flex abrasion cycles required to rupture fabric x (a naturally aged cellulosic textile) after treatment with distilled water, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

1.5 Delimitations

Out of necessity this study has used cotton which has been aged by accelerated means as well as naturally aged cotton and unaged cotton. Cotton aged by accelerated means is more uniform than naturally aged cotton. Accelerated ageing also provides a means of studying deterioration of textiles in a convenient time frame. It provides for an unlimited supply of fabric which is important as expendable historic fabric is rare. However, many unknown or complex interactions occurring in natural ageing may be eliminated when ageing is accomplished by accelerated means.

For the most part, cotton fabric was used as a representative cellulosic textile, although its reactions are not identical to those of rayon or linen. In addition, one fabric construction, a plain weave print cloth was used in all experiments involving new fabric. For practical purposes it was not possible to use fabric of varying yarn construction and weave. These factors do influence physical properties which are evidenced in such testing methods as used for abrasion. The penetration of the deacidification solutions does differ in fabrics of varying constructions.

Finally, these methods of deacidification apply only to cellulosic products, therefore methods of reducing the degradation of other fibre types such as wool and silk were not addressed.

1.6 Definitions

For the purpose of this study the following definitions apply:

Neutralization - a process of reducing the acidity and attaining a neutral pH of 7.

Deacidification - a process which brings about neutralization of the textile, and leaves an alkaline reserve in place.

2. LITERATURE REVIEW

It is the intent of this review to outline the structure of cellulose and the manner in which it is degraded by various agents. Cellulose is the major component of a number of fibres including flax, cotton and rayon, thus knowledge of its structure and reactions is helpful in understanding how these fibres deteriorate with age and use. The specific morphologies of cotton, flax and rayon will not be included here because there are many excellent resources providing this information (deGruy, Carra and Goynes, 1973; Hearle and Peters, 1963; Mauersberger, 1952; Shenouda, 1979).

This literature review encompasses all cellulosic fibres used in the construction of textiles. Much of the information available pertains to cotton cellulose, because cotton provides the purest form of cellulose for study and allows examination to take place with the least interference from impurities. The information derived from the study of cotton cellulose also pertains to flax and rayon, although it must be recognized that the impurities in these fibres and differences in arrangement of the molecules will change the chemistry of these fibres somewhat and hence the rate and extent of many reactions.

2.1 Cellulose

Cellulose is a common molecular compound encountered in many forms. Nature has used it as the primary ingredient in plant tissues, where it is especially useful in stems as supporting tissues. As such, there are various sources of cellulose which can be used as textile fibres, including bast fibres, such as linen, jute, ramie and hemp, leaf fibres, such as abaca and sisal, and seed hairs, including cotton and kapok (Hearle and Peters, 1963).

2.1.1 Polymer Formation

Cellulose is a polysaccharide, belonging to a larger class known as carbohydrates. It is a polymer made up of many glucose units.

Glucose is a monosaccharide containing six carbon atoms, as shown in Figure 2.1.

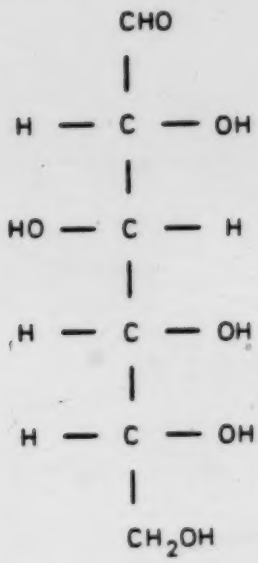


Figure 2.1 Glucose Molecule

This is the D-glucose isomer (as opposed to the L-isomer found elsewhere in nature) referring to the absolute configuration of the hydroxyl groups on C-5. The glucose found in the cellulose molecule is a cyclical structure which may be called a hemiacetal because of the intramolecular reaction of the hydroxyl group at C-5 with the aldehyde group at C-1. The result is the configuration illustrated in Figure 2.2. In this form, the pyranose form, C-1 is assymmetric and the form pertinant to the cellulose structure is the β-configuration with the hydroxyl in the equatorial position. The ring assumes the chair conformation (see Figure 2.3). This is the conformation of least energy as it provides the least amount of interaction among the hydroxyl groups.

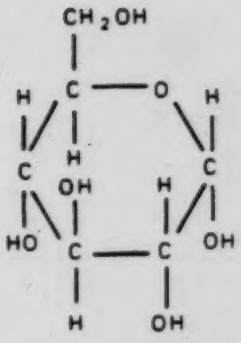


Figure 2.2 Cyclical D-glucose molecule (after Peters, 1963)

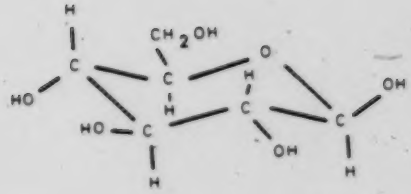


Figure 2.3 The chair conformation of D-glucose

The cellulose molecule is made up of these β-D-glucose molecules linked together by an oxygen bridge at the C-1 and C-4 positions. This oxygen bridge, termed the 1,4-β-glycosidic bond, is formed through the elimination of water between two hydroxyl groups. Two glucose units joined together are often shown as the repeat unit of cellulose as it indicates the 180° rotation of the one unit in relation to the other when forming the bond. Each repeating unit contains two anhydroglucose units, that is, glucose in which water has been eliminated in forming the oxygen bridge. The linear polymer is illustrated in Figure 2.4.

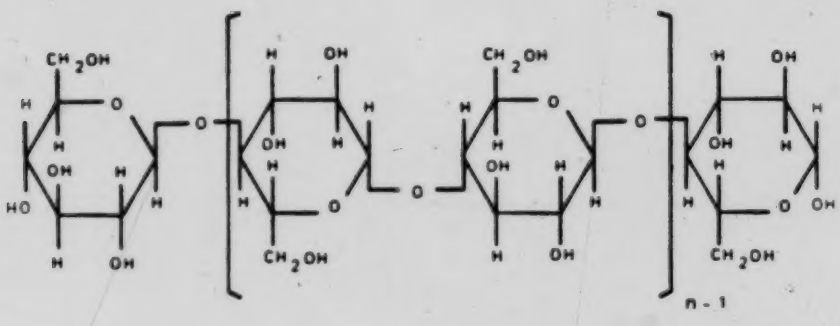


Figure 2.4 The cellulose polymer illustrating the two anhydroglucose units as the repeat unit

(after Peters, 1963)

The degree of polymerization (D.P.) refers to the number of glucose units bridged together to form the cellulose molecule. Cotton cellulose that has not suffered damage of any kind may have a D.P. as high as 10,000 units when measured by the ultra-centrifuge technique. Flax is known to have a D.P. of about 36,000 units (Peters, 1963). The D.P. is an important consideration as it has direct consequences on the physical properties of the fibres such as strength and flexibility.

The chemical formula for cellulose can be summarized as follows:

$$\{ C_6H_{11}O_5 \cdot (C_6H_{10}O_5)_n \cdot C_6H_{11}O_6 \}$$

where n = D.P. (Shenouda, 1979).

In Figure 2.5 it can be seen that each glucose unit contributes three free hydroxyl groups as sites for reactivity. One hydroxyl group is classified as being primary; it is attached to a carbon atom which is linked to one other carbon. The other two are secondary, because the carbon atom, to which the hydroxyl group is attached, is linked to two other carbon atoms (Peters, 1963; Solomons, 1976) (see Figure 2.5). The glycosidic link also presents a potentially reactive site.

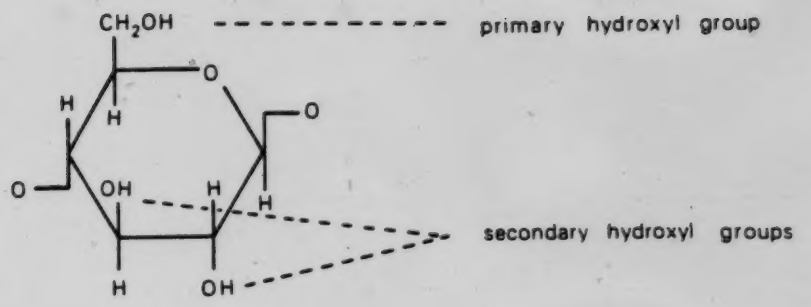


Figure 2.5 The anhydroglucose unit showing the designation of the hydroxyl groups

Each cellulose chain has two distinct ends, one a non-reducing end and the other a reducing end (Figure 2.6). The reducing end is so-called because of the potential formation of an aldehyde group. The C-1 hydroxyl group can react with the ring oxygen atom adjacent to it, causing the ring to open and a new aldehyde group to form. The formation of an aldehyde group increases the reactivity of the cellulose fibre. Therefore, the cellulose with the lower D.P. has the higher potential to react.

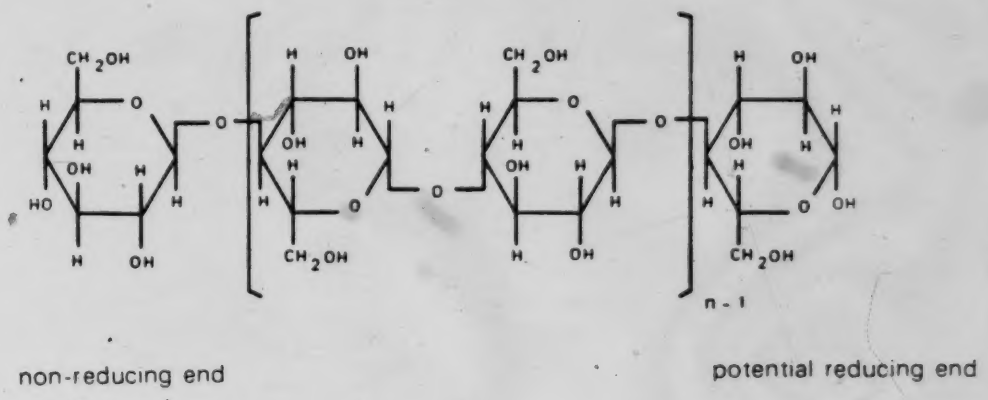


Figure 2.6 Representation of a cellulose polymer showing the non-reducing end and reducing or potential aldehyde end

2.1.2 Fibre Morphology

The molecules in cellulose are arranged into fibrils which are further grouped into fibres. There are areas of crystallinity in which the molecules within the fibril and fibrils are closely associated through hydrogen bonding. These areas are fairly resistant to chemical activity because the molecules are closely associated and there are strong attractive forces (hydrogen bonds). The crystallites are interspaced with amorphous areas where the molecules are loosely associated and poorly organized. In the amorphous areas there is the potential for reactivity as not all of the reactive sites are tied up in hydrogen bonds. One important factor therefore in a fibre's propensity to react is the amount of crystalline material as compared to its amorphous material.

2.1.3 Fibre Reactivity

Factors which determine the stability of the cellulose fibre can be summarized as the degree of polymerization, the ratio of crystalline to amorphous areas, whether the fibre is swollen and the nature of the swelling agent. Polar liquids cause swelling of cellulose and increase the accessibility of the hydroxyl groups, thus increasing the reactivity.

The various types of cellulosic fibres have different levels of purity. Cellulose is the major component of the fibres, however, there may also be lignin, waxes, pectins, other sugars and hemicelluloses present. These affect the stability of the cellulose and, in fact, determine the rate of deterioration. Cotton is the most pure form of cellulose containing 88-96% cellulose (Shenouda, 1979). Flax has about 60-85% cellulose, the remainder being waxes, hemicelluloses and lignin (Peters, 1963). Hemicelluloses enhance reactivity as they contain even more hydroxyl groups than cellulose.

2.2 Chemical Reactions

There are two main reactions involved during the degradation of cellulose, oxidation and hydrolysis. All factors affecting degradation, such as the amount of moisture present, the impurities within the cellulose, the condition of the cellulose, the temperature, the radiation and the pollution levels to which the cellulose is exposed, cause either oxidation or hydrolysis.

2.2.1 Oxidation

All organic materials undergo degradation of an oxidative nature. Oxidation is the reaction of an organic compound with an agent, such that a loss of electrons occurs (Waters, 1964). Autooxidation, or self-oxidation, is of particular importance as it refers to the slow oxidation which takes place in free oxygen, thus in the air that we breathe, and at moderate temperatures (Waters, 1964). Autooxidation is promoted by light and the presence of small quantities of catalysts, such as the impurities found in cellulose, various peroxidic substances, dyes and mordants. Oxidation, generally, is not as harmful as hydrolysis, however, it leaves the cellulose more sensitive to hydrolysis (Rånby, 1959) or to degradation from subsequent exposure to alkalies (Richards, 1971). It is accepted that oxidation causes a complex series of reactions in cellulose, all of which are not completely understood. These reactions are inter-related and ultimately bring about the degradation of the artefact (Feller, 1977).

The oxidation may occur at any one of several sites on the anhydroglucose ring, most likely in an amorphous region. The products of oxidation depend on the type of hydroxyl, whether primary or secondary, undergoing the reaction. Oxidation of the primary hydroxyl group at C-6 produces an aldehyde group which is easily oxidized further to a carboxyl group (Figure 2.7). In this event, there is no decrease in fibre strength as the cellulose chain has not been broken. With the production of carboxyl groups, there is an increase in the level of acidity. Oxidation at C-2 and/or C-3 may lead to the development of ketone groups or aldehyde groups (Figure 2.8). In the latter situation the anhydroglucose ring is opened, making the ring susceptible to cleavage at the oxygen link, and thus breaking the main chain. The

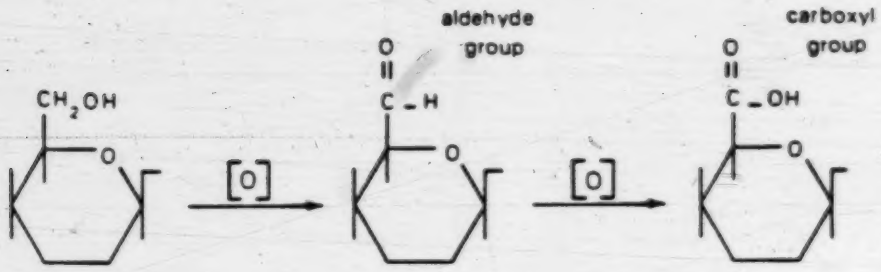


Figure 2.7 The oxidation reaction at C-6

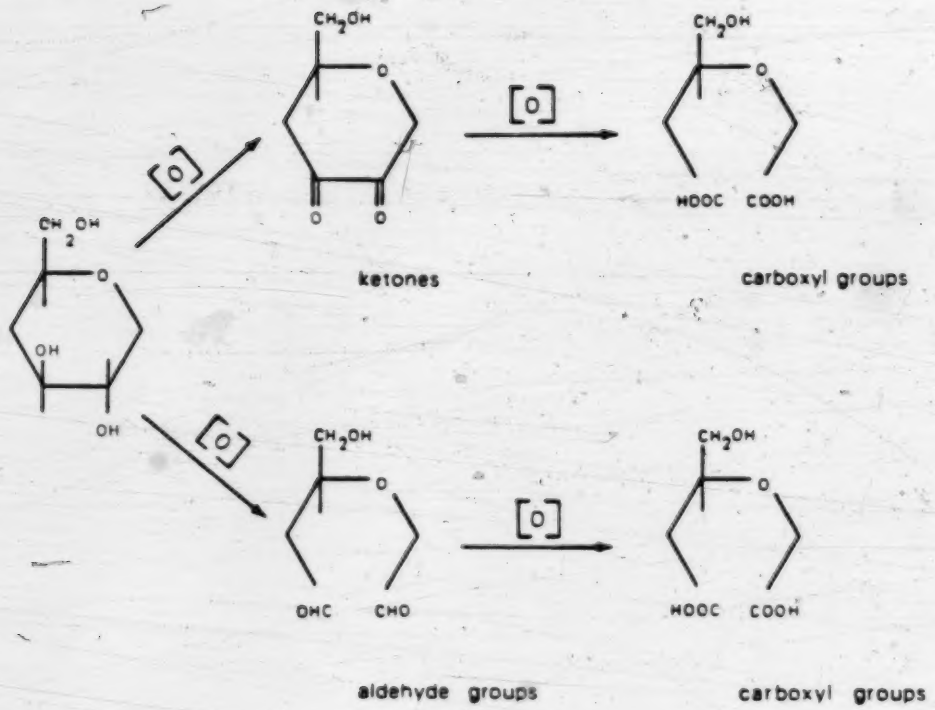


Figure 2.8 The oxidation reaction at C-2 and C-3

aldehyde and ketone groups may be further oxidized to carboxyl groups (Peters, 1963) albeit that the aldehydes are more susceptible to further attack than are ketones (Waters, 1964). The increase in carboxyl groups again increases the level of acidity.

For a reaction to take place enough energy must be supplied to break the chemical bond. This necessary energy level is referred to as the activation energy. Heat is one source of energy which increases the rate of chemical deterioration. At any given temperature there will be some molecules which possess the activation energy necessary for a reaction to occur. If a cellulosic textile is kept at room temperature, whether on display or in storage, the effect of temperature alone in causing degradation, although not eliminated, will be minimal. Of all agents causing damage heat is the least deleterious from this standpoint (Thomson,1978).

Another means of attaining the activation energy required by a molecule to permit a reaction is through the absorption of light energy or visible radiation. Light is emitted by a stream of particles, or photons, travelling in a wave-like pattern. The wavelength of the radiation depends on the amount of energy associated with the photons (Scott, 1965). The energy of the photons within the ultraviolet region of the spectrum is enough to break the bonds of functional groups such as hydroxyl or carboxyl groups occurring in cellulose (Scott, 1965). Thus the energy can be transferred from the photon to the bond resulting in a broken bond. This produces free radicals which allow the process of oxidation to continue. The effects of heat and light are further accelerated in the presence of oxygen and moisture.

Colour change in a cellulosic textile is visual evidence that the cellulose has been oxidized. Colour change (yellowing) in organic molecules is usually associated with the development of unsaturation, particularly conjugated double bonds. It has been shown that if cellulose contains many aldehyde groups, then upon exposure to alkalies, or further oxidation, degradation products, such as furan derivatives, are formed. The formation of these condensed furan derivatives causes yellowing (Hernádi, 1976; Lewin, 1965).

Therefore, because the degree of polymerization is not decreased during oxidation, oxycelluloses, the products of oxidation, may have the same strength as the original cellulose

molecule. Due to an increased number of carboxyl groups there is an increased level of acidity. This increase in acidic groups may increase the susceptibility of the glycosidic bond near the carboxyl group to hydrolyze (Davidson and Nevell, 1956; Davidson and Standing, 1951). Oxidized cellulose becomes sensitive to subsequent exposure to alkali. Due to the increased number of aldehyde groups there is a corresponding tendency for white cellulosics to yellow.

2.2.2 Hydrolysis

A direct consequence of hydrolysis is the breaking of the glycosidic bond. The effects of this reaction are more damaging and apparent than those of oxidation. The fibres lose strength and ultimately become a powder.

Hydrolysis occurs in the presence of acids. The rapidity of the reaction depends, among other things, on the concentration of the acid (Roberson, 1976). The breaking of the 1,4-glycosidic link can occur anywhere along the length of the cellulose chain, however, the amorphous regions are more susceptible to hydrolysis than the crystalline regions because acids can penetrate these regions (Peters and Still, 1979). Following the degradation of the amorphous regions the edges of the crystallites become involved in the hydrolytic reactions (Sharples, 1971). Hydrocelluloses are the products of hydrolysis. With each broken glycosidic link, a new reducing group is produced, which in turn is susceptible to oxidation to a carboxyl group (Figure 2.9).

The effects of hydrolysis on a cellulose fibre are many. The degree of polymerization is lessened and thus the tensile strength decreases. The density is increased due to the removal of amorphous regions and, accordingly, there is a decrease in the moisture regain. The cellulose chain is more reactive due to the newly formed aldehyde groups at chain ends and has an increased tendency to oxidize which again increases the acidity level.

From the above discussion, it can be seen that there is a constant interplay between oxidation and hydrolysis, the one perpetrating the other. Parks (1971) found that with the introduction of carbonyl and carboxyl groups onto the molecule through oxidation there is a

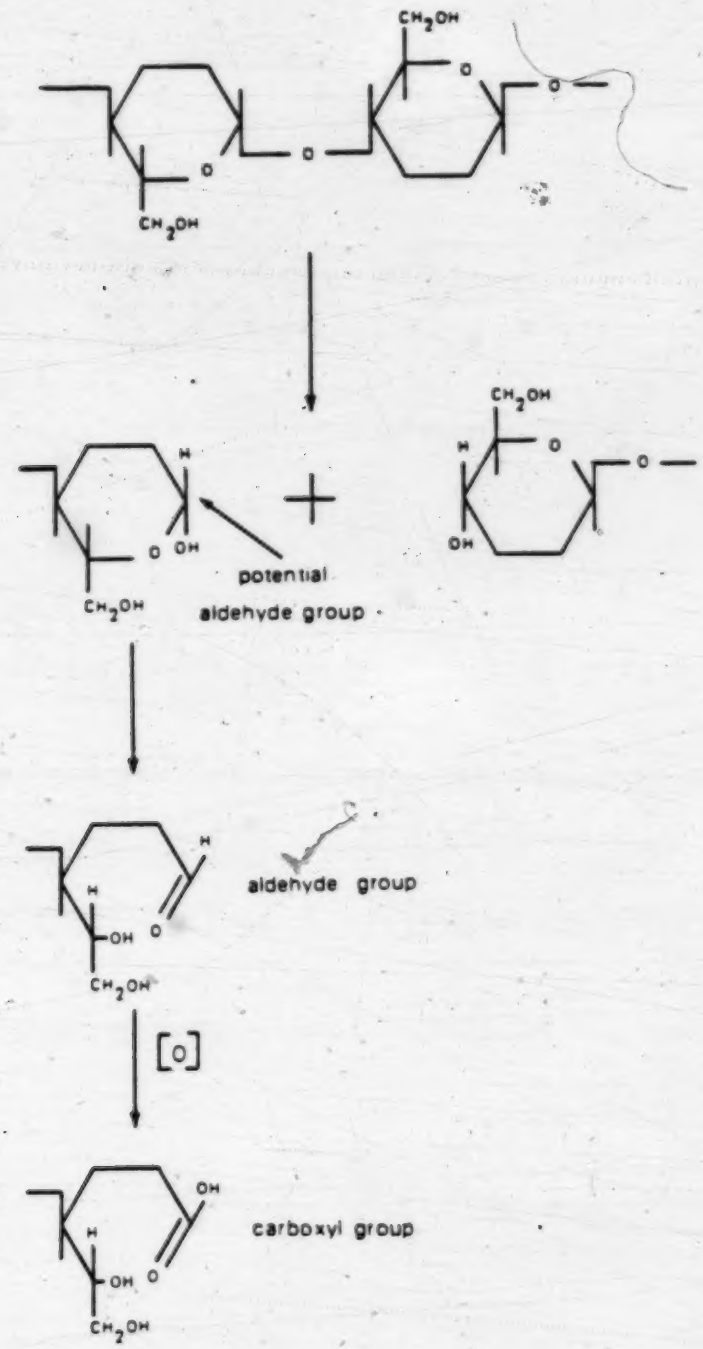


Figure 2.9 The hydrolytic and subsequent oxidation reactions

destabilization of the cellulose. Correspondingly, with the degradation of cellulose by hydrolysis, there is further potential for the development of these functional groups. The reactions are accelerated by high temperatures, moisture, light and oxygen.

2.2.3 Acids

2.2.3.1 Sources of Acids

Environmental pollution is a serious and major source of acidity. When any fuel is burned, the inherent sulphur combines with the oxygen in the air to produce sulphur dioxide. This is easily converted to sulphur trioxide through the reaction with additional oxygen. Sulphur trioxide attracts moisture and becomes sulphuric acid. The hydration of sulphur trioxide to sulphuric acid can happen in the air or on surfaces which contain moisture such as textiles (Thomson, 1978). Treatments to cotton during and after its manufacture leave acidic groups present in the textile. This includes the residual products of dyes, mordants and bleaching. If during storage or exhibition a fabric rests on untreated wood or paper, the fabric again comes into contact with acids. Micro-organisms, for example fungi and bacteria, produce enzymes which are acidic (Howard and McCord, 1959). Degradative products of oxidation are acidic and aid in hydrolytic reactions (Rånby, 1959). Davidson and Standing (1951) concluded that the presence of the carboxyl groups enhances the susceptibility of the glycosidic linkage near the carboxyl group to hydrolyze.

2.2.3.2 Effects of Acids

As indicated above, acids cause the rupture of the cellulose chain by cleaving the 1,4-glycosidic bonds, eventually yielding a mixture of short chain molecules, including glucose, at a levelling off D.P. of about 200. The extent of the hydrolytic reaction depends on the nature of the acid and its concentration. The accessibility of the cellulose to the reactant, that is, the ability of the acidic solution to diffuse into the cellulose molecule, is also a factor affecting the extent of deterioration.

2.2.4 Alkalies

2.2.4.1 Sources of Alkalies

There is a variety of potential sources for alkalies, however, this study is only interested in the ramifications of using an alkaline buffer. Therefore, other sources will not be discussed but rather, the effects of alkalies on cellulose will be explained.

2.2.4.2 Effects of Alkalies

Alkalies generally do not affect cellulose unless the cellulose is in an oxidized state or the temperature and concentration of the alkali are high. This statement can serve as a warning because cellulose which has been oxidized may not show any signs of physical weakness, however, any subsequent exposure to an alkali may cause scission of the chain at the point of oxidation (Richards, 1971).

Autooxidation:

Cellulose which is pure and neutral is stable in air at room temperature. By increasing the temperature and humidity levels the oxidation of the cellulose by atmospheric oxygen increases. Autooxidation, that is, the oxidation of the hydroxyl groups by molecular oxygen, is catalyzed under alkaline conditions, thus making alkali cellulose susceptible to oxidation and subsequent depolymerization. It has been suggested that this is a chain reaction propagated by free radicals (Entwistle, 1949a & b). Under alkaline conditions, Sinkey (1973) postulates that the peroxides produced by reaction with the molecular oxygen are the source of free radicals, hydrogen free radicals being the most likely.

β-alkoxy-elimination:

Any carbonyl-containing oxidized cellulose is sensitive to alkali and liable to chain scission at the cellulose unit which contains the carbonyl group. Alkoxyl or hydroxyl groups in the β-position to an electron withdrawing group, in this case a carbonyl group, are eliminated on subsequent exposure to an alkali base. The presence of an hydrogen atom in the α-position

to these groups is also needed for the scission to take place. Alkoxyl groups are more easily eliminated than are hydroxyl groups (Figure 2.10).

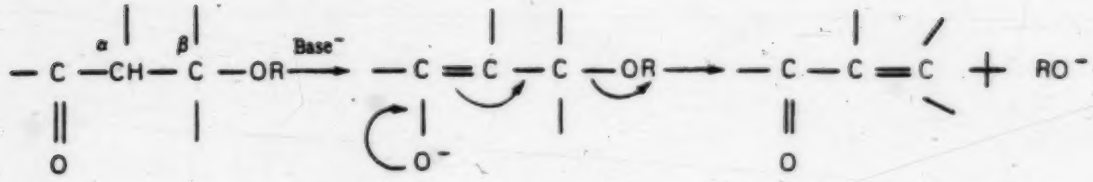


Figure 2.10 Elimination of β-alkoxy groups

(after Richards, 1971)

The carbonyl groups, whether aldehydes or ketones, can be located at various points on the cellulose ring, that is at C-2, C-3 or C-6. This β-alkoxy-elimination reaction, otherwise known as *peeling*, can also occur from the reducing end of the cellulose chain, stripping off one glucose unit at a time and leaving it in a water soluble form. The possible carbonyl sites and the associated alkoxyls are shown in Figure 2.11.

Extensive reviews of this alkaline degradative process can be found in the literature (see Kerr, 1982; Lindberg, Lönngren and Svensson, 1975; Peters and Still, 1979; Richards, 1971) and therefore the actual reactions will not be followed here. Figure 2.12 demonstrates one of the possible reactions with aldehyde groups at positions C-2 and C-3. As illustrated, the products of β-alkoxy-elimination are small molecules. There is also a shortening of the cellulose chain and consequently the cellulose structure is weakened.

β-alkoxy-elimination continues until an alkali-stable molecule is formed (Ziderman and Bel-Ayche, 1978). The stabilization reaction which is brought about by a rearrangement of the reducing end groups competes with the elimination reaction, the elimination rate being greater than the stabilization rate.

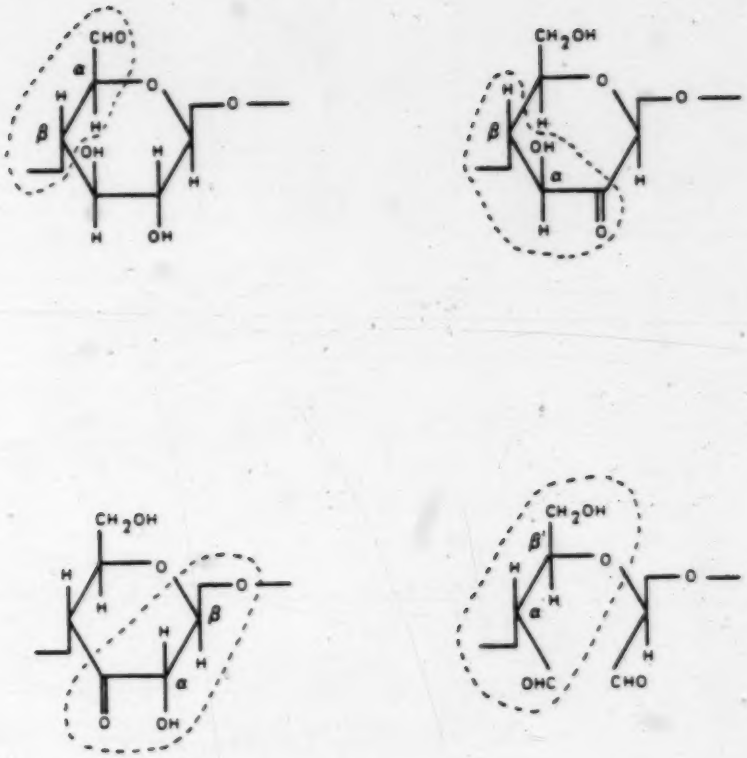


Figure 2.11 β-alkoxycarbonyl groups in carbonyl-containing oxidized cellulose (after Richards, 1971)

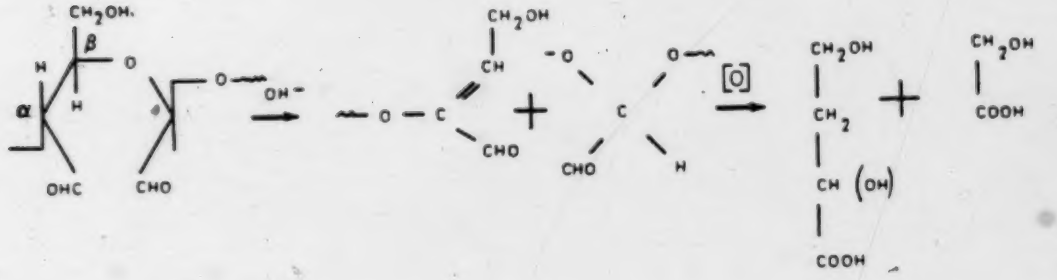


Figure 2.12 Alkaline degradation of carbonyl-containing oxidized cellulose (after Richards, 1971)

2.3 Degradation

From the above discussion, it is evident that textiles deteriorate with time. The extent of this deterioration can be measured as changes in certain physical properties, such as strength, appearance and hand, and colour. Certain chemical properties are altered as well. There is a decrease in molecular weight and degree of polymerization and, consequently, an increase in solubility and fluidity of cellulose solutions. There is an increase in the ratio of crystalline to amorphous material in fibres due to the loss of amorphous regions as these are the initial sites of degradation. Thus the density of the cellulose increases. Moisture regain decreases because the amorphous areas are lost. Naturally, the amount of oxidized cellulose increases with time and therefore an increase in the number of carbonyl groups can be expected. There is usually a decrease in the pH level due to an increase in the number of carboxyl groups (Berry, Hersh, Tucker and Walsh, 1977).

2.4 Use of Deacidifying Agents

There is ample evidence of improved permanence of paper after treatment with alkaline agents. This has culminated in the work by Smith (1970) in his development of the Wei T'o® solution for the non-aqueous treatment of library and archival papers. Others (Arney, Jacobs

and Newman, 1979, 1980; Tang, 1981; Wilson, 1981) have studied the effect of aqueous deacidification solutions on papers and concluded that the treatments are beneficial in that they increase the lifetime of papers. Arney *et al.* (1980) conclude that both oxygen-dependent and independent degradation processes are retarded with the application of calcium carbonate on paper although the relative importance of each reaction changes when the paper is buffered. They concede, however, that the deacidifying treatments used on paper artefacts alter the deterioration process in ways which are not understood and that there remain many questions to be answered.

Ideally, a deacidification solution should have a number of characteristics. It should be safe to use from a human health standpoint. It should deacidify the textile to a neutral pH and impart an alkaline reserve to combat future acidity. Since the alkaline buffer is expected to remain on the substrate for an extended period of time, it should have the capability of remaining inert. Enough alkaline reserve must be left in place to neutralize both internally and externally derived acids.

2.4.1 Indications for Use

The pH measurements of naturally aged textiles, stored in museums and historic houses under varying conditions, have been taken to illustrate that these textiles are at considerable risk to hydrolytic attack (Dawson, 1980; Peacock, 1980). In fact, Dawson (1980) found that many of the samples she collected in her study conducted in England, contained sulphuric acid presumably due to exposure to atmospheric sulphur dioxide. The measuring of pH levels on textiles remains rather tenuous however due to the method which must be employed. Only the use of the surface flat-headed electrode is appropriate for historic textiles as it is not conceivable to destroy these textiles for the purpose of taking a pH measurement. In fact, Hackney and Hedley (1981) found that pH readings can only be used as a guide to the condition of a textile and that the pH levels depend on the predominant mechanism of deterioration that the textile has undergone.

Many of the acidic products, whether ions or small molecules released from the degraded cellulose, are water soluble. However, any assumption that all acids have been removed by washing must be questioned. In fact, studies by Ant-Wuorinen and Visapää (1963) show that the last traces of acids are very difficult to remove from cellulose. These acids that remain can promote hydrolysis, and the degradative process is in motion once again. Hey (1979) underlined this problem with respect to traces of acid left in paper articles when she wrote,

> "Washing and deacidification are the two most fundamentally important stages of paper's conservation. Whatever else may be done in the way of repair, resewing, rebinding, remounting, it is of only *cosmetic* value if the paper itself has not been left in the healthiest possible condition." (p. 66)

There is no reason to believe that cellulosic textiles would behave any differently than paper in this regard.

Washing remains a routine procedure in most textile conservation laboratories. The principle behind this operation has always involved the use of the ***purest*** form of water for the washing. Thus, the use of deionized or distilled water has always been advised. This may have been precipitous as studies by Tang and Jones (1979) show that purified water used for washing papers actually shortened the life of these papers when they were later subjected to accelerated ageing. Theoretically, the washing process may be seen as leaving the cellulose in a more reactive state, as successive rinsing in deionized or distilled water actually removes any salts which have formed naturally and protonates the carboxyl groups. Tang and Jones (1979) found that rinse water passed over a column of calcium carbonate chips was advantageous in that a small amount of calcium carbonate, dissolved in the rinse water, was transferred to the paper. This salt reduced the number of carboxyl groups in the free acid form and reduced the reactivity of the paper. They observed that paper washed in tap water often performed better upon accelerated ageing than paper washed and rinsed in distilled water. They were quick to point out, however, that the use of tap water is not adviseable in the light of other ions it may contain. This discussion points out the fact that washing is not enough to protect cellulose, in fact, it may be detrimental if carboxyl groups are left in the free acid form. Moreover, Tang

(1981) suggests the possibility of washing and depositing the alkaline buffer in the same treatment procedure.

2.4.2 Justification for Use

It is difficult to justify the use of alkaline buffers on cotton fabric when much research regarding the effectiveness of these buffers has been done on paper. Some studies, however, stand out in their relevance to textiles. One such study (Davidson and Nevell, 1956) clearly points to the enhanced stability of cellulose which can be produced by neutralizing the carboxyl groups of oxycelluloses. Davidson and Nevell treated the oxycelluloses with sodium acetate, thus tieing up the acidic groups as sodium salts. These groups were more stable in the salt form than they were in the free acid form.

Although there is a considerable body of research on paper deacidification there is a scarcity of material related specifically to cellulosic textiles. In addition, the majority of the research on paper relies solely on measurements of physical changes to the fabrics upon ageing in order to evaluate the effectiveness of the alkaline buffers.

Peacock (1983), with her work on linen textiles, evaluated deionized water and two deacidifying agents, magnesium bicarbonate and MMMC (Wei T'o® solution #2). Linen fabrics were dipped or sprayed, then after moist oven ageing and acid hydrolysis ageing used in combination, changes in weight, pH, colour, stiffness and tensile strength were measured. The presence of the alkaline buffers benefitted the textiles in that they experienced decreased rates of weight loss, and minimal colour change, however, the stiffness increased. She also found that the method of application, whether dipped or sprayed, had an effect on the degree of protection offered to the linen textile. Her results indicate that immersion of the textiles in an aqueous deacidificant provided better protection than did spraying of the same agent and that spraying of nonaqueous treatments was better protection against degradation than was dipping. She offers no explanation for these results.

Kerr's work (1982) is reinforced by these findings. She set out to investigate the effects of several alkaline buffers on cotton fabric. In addition, it was hoped that the mode of degradation, whether oxidative or hydrolytic, would be discovered when alkaline-buffered cotton was subjected to accelerated ageing. To this end, chemical changes as well as physical changes to cotton fabric upon ageing were investigated in conjunction with the use of three deacidifying agents, namely, calcium hydroxide, magnesium bicarbonate and MMMC (Wei T'o® solution #2). The chemical changes were monitored by measuring changes in intrinsic viscosity, carbonyl content and carboxyl content. Her conclusions point to the beneficial effects of using deacidifying agents to slow down the rate of degradation. From her research it is possible to conclude that the agents, because of their alkalinity, are able to neutralize internally-generated acids as well as those absorbed from external sources. In addition, she was able to show that the agents actually reduce the rate of oxidation of the cellulose.

Block (1982) also illustrated, through the measurement of changes in tear strength upon ageing, that the presence of calcium carbonate on new fabrics protected the fabrics to a certain extent from degradation upon ageing. This finding was subsequently challenged by measurements of colour change of treated new fabrics upon ageing (Block, 1982). It is worth noting that the apparent lack of difference in the rate of colour change upon ageing for treated and untreated cotton conflicts with findings of Kerr (1982).

2.4.3 Kinds of Deacidifying Agents

As the purpose of treating the textiles is to neutralize the pH of the fabric and possibly leave an alkaline residue, it would seem logical to look to the alkaline elements as possible agents. Varying alkaline materials do behave differently when applied to the cellulose substrate. Using salts of alkali metals, such as sodium carbonate, when treating paper, darkens the colour of paper. This, of course, is a manifestation of oxidation, hence degradation (Williams, Fowler, Lyon and Merrill, 1977). Alkaline earth metals do not have this deleterious effect and it has long been recognized that the divalent alkalies, such as calcium or magnesium, exhibit

positive results when used as buffering agents (Williams *et al.*, 1977).

Hey (1979) in her review of the reaction of cellulose with various deacidifying agents has found a marked difference in the response of cellulose to monovalent hydroxides as compared to divalent hydroxides. Oxidized cellulose is much more prone to the alkaline *peeling* reaction with exposure to monovalent alkalies than with divalent alkalies. Upon contact with divalent alkaline compounds, such as calcium and magnesium compounds, the *stopping* reaction is favoured. Therefore, it seems that the alkaline earth metals are regarded as the preferable agents for use as deacidifying agents (Smith, 1970).

Kerr (1982) in her investigation of the effectiveness of varying agents commonly used to deacidify paper, found that magnesium-based compounds showed the most promise in comparison to calcium-based compounds. Kelly's (1977) findings are in agreement with this as he was able to state that the magnesum compounds are better agents to use for deacidification because they tend to be more soluble than calcium-based compounds and this makes their use more convenient. Their use is disputed by Hey (1979). She favours the use of calcium hydroxide on paper for several reasons. She states that calcium is more readily attracted by the cellulose molecule, thus, a lower concentration of the calcium solution is needed to acquire the same alkalinity within the cellulose. In addition, she indicates that due to the poor attractive forces between cellulose and magnesium, magnesium is carried to the surface by water as the paper dries. She states that magnesium bicarbonate dries as crystals on the surface, producing a *gritting* effect. Wilson, Golding, MacClaren and Gear (1981) countered this by stating that care should be taken to maintain the carbon dioxide content of the solution. If this is done, gritting should not be a problem. Hey further states that magnesium bicarbonate has a lower pH than calcium hydroxide and therefore only neutralizes the stronger acids. Calcium hydroxide neutralizes all acidic groups regardless of how weak. However, it is important to note that either agent neutralizes carboxylic acid effectively and it is carboxylic acid that the conservator is largely concerned about. Magnesium carbonate, as purchased, often has iron as an impurity, which might catalyze degradation. Therefore the purest possible grade of magnesium carbonate

should be used or, preferably, magnesium hydroxide. Although Hey states that calcium solutions are much easier to prepare than are magnesium solutions, the handbook prepared by Wilson, McKiel, Gear and MacClaren (1979) offers simplified methods for the preparation of magnesium bicarbonate solutions.

These problems have not been cited by all researchers; in fact, Williams *et al.* (1977) found magnesium to be a more effective stabilizer of paper than calcium in the presence of a copper catalyst. Kerr, Hersh, Tucker and Berry (1979) agreed that magnesium carbonate is more difficult to apply than calcium hydroxide because of the lower solubility in water and recommended several applications of the buffering solutions to the textiles to achieve sufficient add-on. However, they also found that the calcium hydroxide adversely affects fabric hand and is less effective in preventing yellowing during accelerated aging than magnesium carbonate. Suffice it to say, therefore, that there is evidence of improved permanence of textiles and paper through the use of calcium-based solutions (Arney *et al.*, 1979, 1980; Block, 1982; Kerr, 1982) and magnesium-based solutions (Kerr, 1982; Williams *et al.*, 1977).

Daniels (1982) studied changes in colour of water colour pigments applied to paper during deacidification. The problem of colour change is also of relevance to textiles as a variety of dyes or pigments are applied to textiles for decorative purposes. He found that calcium hydroxide produces an appreciable colour change in the organic pigments he studied, whereas calcium and magnesium bicarbonate caused the least change in colour.

Calcium hydroxide, magnesium bicarbonate and calcium bicarbonate are not stable in air, but are quickly changed to their carbonates, which subsequently form salts when complexing with the acidic groups in the cellulose. This is accomplished by replacing the hydrogen of the acidic group with the metallic ion, effectively neutralizing the acidity.

2.4.4 Aqueous versus Nonaqueous Treatments

The two deacidifying agents found to be the most beneficial from Kerr's (1982) study are methoxy magnesium methyl carbonate (Wei T'o®) and magnesium bicarbonate. They

complement each other well as the latter one is applied from an aqueous solution and the former from a nonaqueous solution, thus presenting an option to the conservator.

Ordinarily, for conservation purposes, the availability of an aqueous system is adequate as an all-purpose cleaning medium. It is also convenient in that the water washes away soluble acids and degradation products in the cellulose. However, textiles often contain water-soluble dyes or finishes or water-sensitive embellishments which must be preserved. In these cases, an alternative to the aqueous system is helpful. Organic solvents do not cause swelling of cotton fibres and thus cause few detrimental effects such as shrinkage and wrinkling. Smith (1970) has successfully devised several products, marketed under the trade name of Wei T'o® for the nonaqueous treatment of books. Solution #2 has been found to be effective in conjunction with cellulosic textiles as well as paper (Kerr, 1982).

2.4.5 Methods of Protection

Reduction in the Rate of Oxidation:

Arney *et al.* (1979) studied the effect of the alkaline buffer on rag paper and newsprint. They determined previously (Arney and Jacobs, 1979) that the degradation of cellulose could be subdivided into that caused by atmospheric oxygen and that caused by an oxygen-independent process. In the subsequent study they were able to establish that both the oxygen-independent and atmospheric process are retarded by deacidification. They concluded that atmospheric oxidation is the primary contributor to degradation when an alkaline buffer is present, and furthermore, that the rate of the oxidation is depressed. This conclusion was substantiated by Kerr (1982). She found that the presence of the alkaline buffers actually decreased the rate of oxidation of cotton fabric during accelerated aging.

Protection Against β-alkoxy-elimination:

It seems from a review of literature regarding work done on pure cellulose that divalent hydroxides encourage the *stopping* reaction necessary for the β-alkoxy-elimination to come to a

halt (Hey, 1979). Chelation of carbohydrates with metal ions, in this case magnesium, has been postulated as the means by which β-alkoxy-elimination ceases (Defaye and Gadelle, 1974). Two glucose rings are bound together through a coordination bond with the magnesium ion. Prerequisites for this coordination complex to form are the presence of carbonyl groups at C2 and C3 and the presence of metal ions to which the cellulose complexes (see Figure 2.13). In this way the *peeling* reaction is blocked.

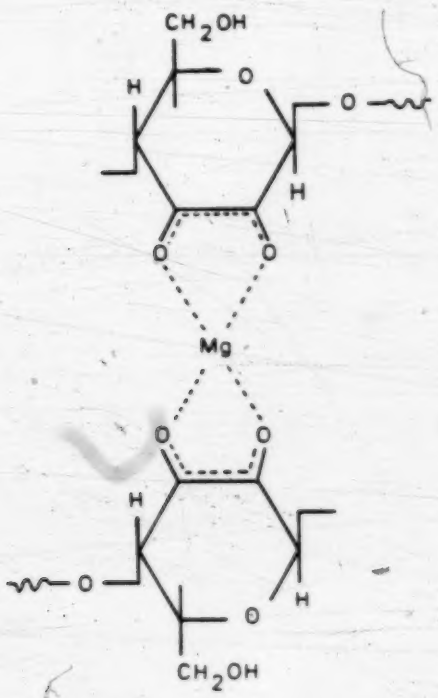


Figure 2.13 The chelated cellulose molecule
(after Defaye and Gadelle, 1974)

Protection Against Autooxidation:

The most important reactions in autooxidation of cellulose are those producing free radicals, presumably from the peroxides which have formed as a result of oxidation. Sinkey (1973) concluded from his research that trace metal ions catalyze the decomposition of the peroxides to create the free radicals. It is postulated that the presence of magnesium deactivates the ions of any transition metals present. This conclusion corroborates the findings of Gilbert, Pavlovova and Rapson (1973), although they stress the fact that it is difficult to conclude the method by which deactivation occurs and the resultant structure of the magnesium-transition metal complex.

2.4.6 Limitations of the Deacidification Treatment

It seems that the presence of the alkaline buffer within the cellulosic substrate protects the cellulose from the level of deterioration that it would otherwise experience with time. The degradative process will continue, however, the extent of the degradation being dependent on the environmental conditions of storage and the maintenance of alkalinity. It should be emphasized that the deacidifying agent can do nothing to restore properties already lost through degradation. It will not enhance strength or flexibility or reduce the yellowing already present when the buffer is applied. It should, however, slow down the rate of deterioration in the future.

2.5 Concerns Raised by Conservators

To most conservators and curators, the ideal treatment for textiles is one that is reversible. If there is little concrete evidence that complete reversibility is possible, they will hesitate to use the treatment. This question has been raised in regards to the deacidification of cellulosic textiles. Hey (1977) expressed this apprehension when writing of the decreased fold endurance of paper after being treated with an alkaline buffer. She offers several reasons for the decreased strength and suggests that it is due to the deposition of solid particles. These

particles, she hypothesizes, may reduce the interfibre bonding, cause stiffening and thus reduce the flexibility of the fibres in relation to each other. Solid particles abrading and causing breakage of the fibres is another possibility. Any loss of strength or damage due to abrasion is irreversible making the treatment unacceptable. These concerns have been extended to textile fibres as well.

Padfield (1982) is worried about the hygroscopic nature of carbonate salts which deliquesce at high relative humidities leaving the textile in close proximity to moisture. The environment around the textile, he suggests, is more chemically reactive due to the presence of moisture than it would be if the buffering salts are not present. He is further concerned that the products from acid attack on carbonates are also hygroscopic. With the increased moisture content, he fears that an electrolytic cell may be established within the fibres (Padfield, Note 1). In any case, these carbonate-loaded artefacts are, as suggested by Padfield (1982) excellent absorbers of atmospheric pollutants especially acidic gases such as sulphur dioxide. The mixture of moisture and pollutants produces sulphuric acid readily which is harmful to cellulosic materials. These concerns may be relevant only in museums which are not environmentally controlled and conditioned.

Lyall (1982) expressed a concern resulting from studies she made with magnesium bicarbonate. She found that during the deacidification of groundwood paper there was an increased yellowing. She concedes, however, that the treated samples after ageing were about the same colour as the non-buffered samples.

Finally, a question has been raised regarding the relevance of using new fabric to evaluate the efficacy of deacidification (Block, 1983). He believes that aged cellulosic textiles have no carboxyl groups left to neutralize as they decay away leaving only the carbonyl groups which continue the degradation process. Thus, he postulates that the treatment is not effective on already naturally aged textiles. Block does not address the problem of acidity produced extrinsically or the development of new carboxyl groups with time.

3. PROCEDURES

3.1 Materials

3.1.1 Test Fabric

New and aged fabric were used to study the effects of the deacidifying agents. The fabric used in the new state and after accelerated ageing was desized, unbleached, 100% cotton, purchased from Testfabrics, Inc., Middlesex, N.J. It had a count of 35 x 31 yarns/cm (warp x weft) and a weight of 117 g/m^2. This fabric was subjected to accelerated ageing by exposure to either a dose of 50 or 75 Mrads of ionizing radiation. Irradiation in an electron accelerator manufactured by High Voltage Engineering Corporation was carried out at North Carolina State University School of Textiles. The effects of ionizing radiation on cotton fabric have been documented by Berry *et al.* (1977). Although the mode of degradation differs from natural ageing, the end result is similar in that the cellulose is both hydrolized and oxidized.

Naturally aged fabrics were also tested to examine their response to alkaline buffers. These 100% cotton fabrics were donated by individuals and by the University of Alberta's Historic Costume and Textiles Study Collection. A minimum number of experiments were carried out on naturally aged fabrics because the yardage was limited and the previous history of each piece of fabric was unknown (see Appendix I). Commercially dyed new fabrics were also used to examine the effects that the deacidifying agents have on the colour of the fabrics.

3.1.2 Deacidification Agents

Magnesium bicarbonate ($Mg(HCO_3)_2$) was made from laboratory grade magnesium hydroxide ($Mg(OH)_2$) using the method indicated by Wilson *et al.* (1979). The source of CO_2 was dry ice.

Methoxy magnesium methyl carbonate (MMMC) was used in the form available commercially as Wei T'o®. The Wei T'o® solutions were solution #2, used for dipping, and

solution #12, used for spraying. Solution #2, used for dipping, contains trichlorotrifluoroethane as the primary solvent and methanol as the secondary solvent for the methoxy magnesium methyl carbonate. Solution #2 was chosen as it is resistant to precipitation caused by water or humidity. The I.U.P.A.C. name for this compound is magnesium methoxy (methyl carbonato - O)- and its formula is $CH_3OMgOCO_2CH_3$ (Kerr,1982). Solution #12, used for spraying, contains ethanol as the primary solvent and trichlorotrifluoroethane as the secondary solvent. CO_2 has been added as the propellant. Solution #12 is least affected by humidity and is recommended for penetrating artefacts. These solutions are available from Wei T'o® Associates, Inc., 21750 Main Street, Unit 27, P.O. Drawer 40, Matteson, Illinois, 60443. Control fabrics were wet out with distilled H_2O.

3.2 Methods

3.2.1 The Application of the Deacidifying Agents

The water-treated control and the aqueous $Mg(HCO_3)_2$ treatment were applied through a dipping procedure in which the samples were immersed for 30 minutes. The Wei T'o® was applied by both dipping and spraying techniques. Peacock (1980) found that the spray application offered better protection against degradation than did immersion of the fabric when treating with a nonaqueous reagent. As specific solutions have been formulated for each type of application method, it was appropriate that each method be studied as to its effectiveness.

The pH measurements were carried out in this study to provide assurance that an alkalinity was indeed established after treatment with the deacidifying agents. In addition, pH measurements were taken of naturally aged textiles to give an indication of their levels of acidity and thus their degree of degradation. This remains true providing the textiles have not been unknowingly washed prior to testing. The cold extraction method, as indicated by test method T509 os-77, was used to establish the hydrogen ion concentration (TAPPI, 1977).

It was found, through preliminary testing, that a minimum length of time was needed to achieve an adequate add-on and an elevated pH (Appendix II). Based on these results, the samples were dipped in the Wei T'o® solution for 10 seconds. The Wei T'o® spray was applied to the fabric as suggested in the Wei T'o® instruction leaflet (Note 2). The fabrics were secured in the vertical position and the solution sprayed in a back and forth motion, until the textile appeared uniformly saturated.

3.2.2 Sampling Procedure

A series of three large blocks (80 x 30 cm warp x weft) were cut across the width of the fabric. Each large block was cut into 8 smaller blocks measuring 20 x 15 cm (warp x weft). This size was sufficient to do all of the physical tests required. The blocks of fabric were assigned at random to one of the four treatments. No specimen was cut within 10 cm of the selvedge. This procedure was used for the new samples as well as those samples which were irradiated before treating and testing. The naturally aged textiles were cut and randomized as much as was possible.

Samples were washed in a 0.1% Orvus solution, an anionic surfactant, in distilled water and rinsed repeatedly before treating. The 50 and 75 Mrad irradiated samples were too fragile to withstand washing and thus were treated directly. The naturally aged textiles were similarly washed, with pH readings taken from the initial water bath (see Appendix I).

3.2.3 Test Procedures

1. Moisture regain was measured according to ASTM Method D-2654-76. The following equation was used:

Moisture Regain, $\% = (I - W) / W \times 100$

where I = initial conditioned weight of the specimen, g,

and W = weight of the oven-dry specimen, g (ASTM, 1983).

bending length and the weight per unit area of the fabric as follows:

Bending length, $c = O/2$

where O = the length of the overhang, cm.

Flexural rigidity, $G = W \times c^3$

where W = the weight per unit area, mg/cm^2.

The overall rigidity, G_0 was calculated as follows:

$G_0 = (G_w G_f)^{1/2}$

where G_w = warp flexural rigidity

and G_f = filling (weft) flexural rigidity (ASTM,1983).

3. Abrasion Resistance was evaluated according to ASTM Method D 3885-80. The end point chosen was the point of rupture and the number of cycles needed to reach this point was recorded. This test method was chosen mainly because a small sample size was required (200 mm x 32 mm). Since naturally aged textiles were tested as well as new cotton only a limited amount of fabric was available.

Due to the fragile nature of the treated fabrics which were naturally aged and the fact that they broke after so few cycles, the numbers were not rounded out as indicated in the ASTM Method D 3885-80. Furthermore, to remain consistent, the actual number of cycles to rupture were recorded for the unaged fabrics as well, rather than as indicated in Method D 3885-80. The bar was used as the abradant for this test in conjunction with 2.27 kg of tension and 0.45 kg of pressure. The samples were conditioned and tested in the standard atmosphere for textiles which is 21 ± 1° C and 65 ± 2% relative humidity.

4. To determine colour change after treatment with the deacidifying agents, colour measurements were made with a Hunterlab Tristimulus Colorimeter, Model D25M-9, as indicated by the AATCC Test Method 153-1978. The measurements were made in the CIE L* a* b* scale (CIELAB) where L*, a*, b* measure the perceptibility of colour. L* measures lightness from black to white; a* measures the degree of redness-greenness; and b* measures the degree of yellowness-blueness. The colour difference between two fabrics having undergone

different treatments is ΔE and the formula is as follows:

$$\Delta E = ((\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2)^{1/2}$$

(Hunterlab Tristimulus Colorimeter (Instruction Manual))

The fabric to be measured was layered increasingly until there was no change in the L*, a* and b* values given by the colorimeter.

The colour difference measurements were related to Table 1 of the AATCC Evaluation Procedure 1 "Gray Scale for Color Change" (see Table 3.1) in order to assign a nominal value to the measurements.

Table 3.1: The colour difference ratings of the Gray Scale for Color Change (GSFS)[1] and equivalents as determined by the Hunterlab Tristimulus Colorimeter (CIELAB units) with their corresponding verbal descriptions of the colour change. [2]

GSFS Rating	Colour Difference (CIELAB units)	Verbal Description[3]
5	0.0±0.2	negligible or no change
4-5	0.8±0.2	
4	1.7±0.3	slightly changed
3-4	2.5±0.35	
3	3.4±0.4	noticeably changed
2-3	4.8±0.5	
2	6.8±0.6	considerably changed
1-2	9.6±0.7	
1	13.6±1.0	much changed

[1]Fastness rating as stated in AATCC Evaluation Procedure 1(1982/83).
[2]Table 3.1 is derived from information presented in Section 2 "Colorfastness", AATCC (1982/83).
[3]Verbal description of degree of alteration in shade and strength.

In addition, a panel of experts in colour evaluation was established to compare the CIELAB measurements of colour change to those changes detectable by the human eye. This was set up in accordance with the AATCC Evaluation Procedure 1 "Gray Scale for Colour

In addition, a panel of experts in colour evaluation was established to compare the CIELAB measurements of colour change to those changes detectable by the human eye. This was set up in accordance with the AATCC Evaluation Procedure 1 "Gray Scale for Colour Change"(AATCC, 1982/83).

5. Fibre damage and deacidificant location were evaluated using scanning electron microscopy and electron dispersive X-ray analysis (EDAX). The samples were examined after treatment and after undergoing 1000 cycles of abrasion on the Brush Pilling Tester. Washed, untreated, test fabric was mounted on the lower disc, with the treated sample on the upper disc.

4. RESULTS AND DISCUSSION

4.1 Statistical Analysis

A oneway analysis of variance was carried out using the SPSSx programme available at the University of Alberta, Edmonton, Canada. The dependent variables, colour change, stiffness, moisture regain and flex-abrasion, were measured against the independent variable, treatment, to see if there was a significant difference among the measured means for each independent variable.

Duncan's Multiple Range Test ($\alpha=0.05$) was carried out to establish which means formed a homogeneous subset. As stated in the SPSSx programme printout (Appendix III), a homogeneous subset is one whose highest and lowest means do not differ by more than the shortest significant range for a subset of that size. The analysis of variance and Duncan's Multiple Range test results can be found in Appendix III. Using these results it is possible to establish the acceptance or rejection of the null hypotheses which are laid out in section 1.4.

4.2 Aqueous Extract Fabric pH

Three deacidifying treatments were used in this study. Two of these treatments, methoxy magnesium methyl carbonate (MMMC) in the dipping formula and MMMC in the spraying formula are commercially available as Wei T'o® solution #2, and solution #12, respectively. The third treatment involved dipping specimens in an aqueous solution of magnesium bicarbonate ($Mg(HCO_3)_2$). Fabrics wet out with distilled water were used as the control samples. It is appropriate to study MMMC as well as $Mg(HCO_3)_2$ as each offers particular advantages to the conservator. Because MMMC is applied from a nonaqueous medium, fluorocarbon solvent plus alcohol, its use is prescribed for textiles which cannot withstand the rigors of a water bath. Its availability in both the dipping and spraying formulae provides additional options for conservators. Some textiles may be too large, may be embellished with objects, or treated with dyes which run in water. For these textiles the

spraying formula is a more likely choice than the dipping formulation. $Mg(HCO_3)_2$ is an aqueous treatment that is suitable for any textile which can withstand dipping in water. Regardless of the solvent base, each treatment deposits $MgCO_3$ on the fabric. Therefore, one might expect there to be an elevated pH level and an increase in the fabric weight. In Table 4.1 the level of pH attained and the corresponding weight increase (% add-on) are reported for new cotton fabric treated with distilled water or each deacidifying agent.

Table 4.1: Aqueous extract pH and weight increase (% add-on) of new cotton fabrics after treatment with deacidifying agents.

Treatment	pH[1] aqueous extract	% Add-on[2]
Water (control)	7.5	
MMMC[3] dip	10.5	4.76
MMMC spray	10.4	2.01
$Mg(HCO_3)_2$ dip	10.5	3.89

[1]Average of 2 samples.
[2]Average of 10 samples.
[3]MMMC is methoxy magnesium methyl carbonate (available commercially as Wei T'o® solution #2 for the dipping formula, solution #12 for the spraying formula).

4.3 Colour

It is well known that the treatment of oxidized cellulose with a hot alkaline solution will cause the fabric to turn yellow. For this reason, conservators fear that white historic textiles, treated with alkaline deacidifying agents, will become yellow. Autooxidation, that is, the oxidation of the hydroxyl groups by molecular oxygen, is catalyzed under alkaline conditions (Entwistle, 1949a; 1949b). Lewin and co-workers (1965) determined that it is the oxidation of

the hydroxyl groups on the cellulose chain to aldehyde groups which causes the yellowing to occur. A change in colour of an artefact which is undergoing treatment is undesireable, of course, as is any increase in the extent of oxidation.

In the discussion of colour change each null hypothesis (in italics) will be stated, followed by the statistical analysis which resulted in the acceptance or rejection of the hypothesis. An attempt will be made to explain the results of colour change and relate them to findings in the literature.

It will be recalled that in addition to measuring colour changes instrumentally with a colorimeter, colour change was also evaluated visually using the Gray Scale for Staining (AATCC, 1983). These results are also recorded in the following tables as useful indicators of the changes which can be perceived by the human eye.

a. There is no significant difference in the colour change of new white cotton textiles after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

From Table 4.2 it can be seen that there is a statistically significant difference in the colour of new cotton after treatment with water or the deacidifying agents, therefore, the null hypothesis is rejected. Comparing the numerical results of colour change (Table 4.2) with the visual evaluation (GSFS), an important observation can be made; that is, the colour changes brought about by the treatments are inconsequential. The MMMC dip and spray caused a negligible change. The somewhat greater change due to the aqueous $Mg(HCO_3)_2$ treatment is also considered unimportant since the fabrics whitened slightly rather than becoming yellower.

These results concur with Peacock's study (1983) in which she found that none of the treatments she carried out visibly altered the colour of the new fabrics. She also found that aqueous $Mg(HCO_3)_2$ produced the greatest change upon application but she did not describe the nature of the change. In addition, she found that the magnitude of this colour change decreased among the various treatments with subsequent ageing of the fabrics.

b. There is no significant difference in the colour change of naturally aged white cellulosic textiles after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

Table 4.2: Mean colour difference (CIELAB units)[1] and Gray Scale for Staining (GSFS) rating of new white cotton fabric after treatment with the deacidifying agents, MMMC[2] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [3]

Method of Evaluation	Treatment		
	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
Colorimeter[4] (CIELAB units)	1.16	0.86	2.15
Gray Scale for Staining[5]	4.5	4.8	4.2[6]

[1]Colour difference is calculated using the colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'o® solution #2 for the dipping formula and solution #12 for the spraying formula).
[3]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[4]Average of 10 samples.
[5]Average of 3 evaluations.
[6]The expert panel described the fabric as being whiter.

The following results should be examined in conjunction with Appendix I, the description of each naturally aged fabric. Because the previous history of each fabric differed, the initial whiteness of these fabrics varied considerably. The null hypothesis can be rejected as there is a significant difference in the colour change of each fabric after treatment with $Mg(HCO_3)_2$ or MMMC (see Table 4.3). In six out of the ten cases, the $Mg(HCO_3)_2$-treated fabrics are significantly different from the other two groups. It is also relevant, however, to note that in seven out of the ten cases, MMMC dipped fabrics and MMMC sprayed fabrics form a homogeneous subset. Samples which show the greatest amount of colour change when deacidified seem to be those which were initially the most yellowed, and generally degraded fabrics. Even these changes, however, can be described as only "slightly noticeable", having

Table 4.3: Mean colour difference (CIELAB units)[1] and Gray Scale for Staining (GSFS) rating of naturally aged white cellulosic textiles after treatment with the deacidifying agents, MMMC[2] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [3]

Sample Number	Method of Evaluation	Treatment		
		$Mg(HCO_3)_2$ dip	MMMC dip	MMMC spray
1	colorimeter[4]	0.35	0.96	1.62
	GSFS[5]	4.7[6]	4.8	4.7
2	colorimeter	0.42	1.97	2.06
	GSFS	4.8	4.5	4.5
3	colorimeter	1.30	2.25	1.53
	GSFS	4.3	4.3	4.2
4	colorimeter	1.70	0.84	1.68
	GSFS	4.5	4.7	4.0
9	colorimeter	1.16	2.12	2.82
	GSFS	4.5	4.0	3.8
11	colorimeter	0.49	0.98	0.88
	GSFS	4.5[6]	4.8	4.7
12	colorimeter	1.25	2.12	2.27
	GSFS	4.3	4.3	4.0
13	colorimeter	0.85	2.90	2.14
	GSFS	4.3	3.7	3.8
16	colorimeter	0.68	1.86	2.06
	GSFS	4.8	4.5	4.5
17	colorimeter	0.51	2.56	2.15
	GSFS	5.0	4.3	4.5

[1]Colour difference is calculated using the colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula.
[3]Homogeneous subsets, as determined by Duncan's Multiple Range Test, are underlined.
[4]Average of 6 readings.
[5]Average of 3 evaluations.
[6]The expert panel verbally described the changes as being whiter.

been assigned an average Gray Scale for Staining rating of 3.5 or greater by the expert panel (see Table 3.1).

The $Mg(HCO_3)_2$ treatment initiated the least amount of colour change. This treatment was often described by the expert panel as having a lightening effect and on fabrics 1 and 11 it caused a definite whitening.

When discussing the deacidification of paper, Lyall (1982) stated unequivocally that the introduction of magnesium to an already aged sample caused further yellowing soon after the treatment. She suggested that this indicated accelerated oxidation. In contrast to her findings, however, it is observed in these experiments that the amount of colour change of naturally aged cotton and linen fabric, both of which are purer forms of cellulose than is paper, is not enough to warrant concern. In any event, Lyall (1982) found that upon subsequent ageing, through exposure to U.V. light, the deacidified samples exhibited the same degree of colour change as the non-deacidified counterparts.

c. There is no significant difference in the colour change of white cotton textiles aged by accelerated means after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

From the results shown in Table 4.4 it is evident that both the MMMC dip and MMMC spray treatments form a homogeneous subset, and do cause a considerable degree of colour change in the irradiated fabric. The null hypothesis is rejected because there is a significant difference in the colour change when comparing the $Mg(HCO_3)_2$ treatment to the MMMC treatments.

The 50 and 75 Mrad irradiated fabric was quite yellow and friable as a result of the γ-irradiation, therefore, samples cut from this fabric were not washed before treatment as were all other samples. It was found that upon saturation of the specimens with the deacidifying solutions a yellow colour migrated across the samples to their edges and also caused a yellowing of the treatment bath. This ultimately resulted in a variability in the colour of the treated specimens. Hutchins (1981) found that γ-irradiated cotton fabrics contain degradation products which are yellow in colour and are water soluble. Thus, in this experiment, the

Table 4.4: Mean colour difference (CIELAB units)[1] and Gray Scale for Staining (GSFS) rating of cotton textiles aged by accelerated means, after treatment with the deacidifying agents, MMMC[2] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [3]

Sample Description	Method of Evaluation	Treatment		
		MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
irradiated -50 Mrad	colorimeter[4]	5.70	5.29	8.29
	GSFS[5]	3.2	3.2	3.0
irradiated -75 Mrad	colorimeter	5.98	6.42	8.85
	GSFS	2.8	3.0	2.3

[1]Colour difference is calculated using the colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'o® solution #2 for the dipping formula and solution #12 as the spraying formula.
[3]Homogeneous subsets, as determined by Duncan's Multiple Range Test, are underlined.
[4]Average of 6 readings.
[5]Average of 3 evaluations.

migrating yellow colour is likely degraded cellulose. Colour variation during deacidification can likely be minimized through the washing of the textiles beforehand. If washing is not a possibility, then, judging from the results, treatment with solvent-based MMMC agent would be the better alternative than aqueous $Mg(HCO_3)_2$.

d. There is no significant difference in the colour change of selected commercially dyed cotton fabrics after treatment with each of the deacidifying agents, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

For the sake of brevity, a separate null hypothesis was not written for each colour. The colour change data for dyed fabric was submitted to an analysis of variance in order to determine whether to accept or reject the null hypothesis. The results of these analyses are shown in Table 4.5 as "accept" or "reject". In general, the mean values for the colour change

Table 4.5: Mean colour difference (CIELAB units)[1] of commercially dyed cotton textiles after treatment with the deacidifying agents, MMMC[2] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [3] [4]

Colour of Sample	Treatment			Null Hypothesis
	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip	
yellow	2.29	2.35	1.06	reject
green	0.70	0.70	0.96	reject
mauve	1.15	0.80	0.82	reject
coral	0.83	0.72	0.91	accept
navy blue	0.75	0.70	0.68	accept
light blue	0.64	0.36	0.76	reject
aqua	1.20	0.57	0.73	reject

[1]Colour difference is calculated using the colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula).
[3]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[4]Average of 10 readings.

of dyed cotton fabrics indicate that the deacidifying treatments have a negligible effect on the colour of these commercially dyed fabrics.

Daniels (1982), who has studied the deacidification of works of art on paper, found that there was a change in the colour of organic pigments with differing conditions of acidity and alkalinity. He favoured the use of magnesium bicarbonate or calcium bicarbonate as these had less effect on colour change than barium and calcium hydroxides. The findings of the

present study concur with Daniel's findings in that magnesium bicarbonate causes minimal change.

Examining the above results it is possible to allay the conservator's fear of colour change providing that fabrics to be deacidified are dyed with the same class of dyes as those used on the fabrics in this study. Some dyes are quite pH-sensitive. Because of the alkalinity of deacidifying agents it might be expected that certain dyed museum textiles would undergo a colour change upon treatment. Although the particular dyes used on the cotton in these experiments are not readily changed especially when treated with aqueous magnesium bicarbonate, it is not possible to generalize about all classes of dyes. If a dyed historic fabric is to be treated, the colours in it should be tested before deacidification.

An additional observation, which is not related specifically to the the null hypothesis, may be made at this time. It is encouraging to note that measurements made with the colorimeter approximate closely the results obtained from the expert panel who visually evaluated the treated specimens using the AATCC Gray Scale for Staining Evaluation (AATCC, 1983). A colorimeter can be use with confidence to measure colour change knowing that it correlates well with the subjective assessments which take into account the human means for evaluating colour.

4.4 Stiffness

The results of stiffness measurements, reported as flexural rigidity (mg.cm) are found in Table 4.6 as well as the percent add-on or weight increase which occurred when specimens were treated with various deacidifying agents or distilled water. In addition, the overall flexural rigidity of specimens after treatment is shown in Table 4.7. The overall flexural rigidity combines the stiffness of warp and weft specimens into one value which facilitates comparison among treatments.

The following null hypothesis for warp and weft specimens was tested.

e. There is no significant difference in the stiffness of warp or weft specimens of new cotton textiles after treatment with distilled water and the deacidifying agents,

Table 4.6: Mean flexural rigidity or stiffness (in mg.cm units) of new cotton fabrics after treatment with distilled water and the deacidifying agents, MMMC[1] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [2]

Direction[3]	Treatment			
	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip	Distilled Water
Warp	1828 ± 391[4]	505 ± 71	266 ± 16	196 ± 17
Weft	618 ± 59	153 ± 20	90 ± 9	63 ± 4
% Add-on	4.76	2.01	3.89	

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula).
[2]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[3]10 warp and weft specimens were tested.
[4]Mean ± 95% confidence intervals.

Table 4.7: Mean overall flexural rigidity (in mg.cm units)[1] of new cotton fabrics after treatment with distilled water and the deacidifying agents, MMMC[2] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [3]

Treatment [4]			
MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip	Distilled Water
1063	278	155	111

[1]Overall flexural rigidity, $G_o = (G_w G_f)^{1/2}$
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula.
[3]No tests of homogeneity could be carried out on this data.
[4]10 warp and weft specimens were tested.

MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

The null hypothesis for both warp and weft specimens is rejected. The application of Duncan's Multiple Range Test to the stiffness data shown in Table 4.6 indicated there is no significant difference in warp and weft fabrics which have been treated with $Mg(HCO_3)_2$ and those that have merely been dipped in distilled water. Wilson *et al.* (1981) in their study of the deacidification of paper with $Mg(HCO_3)_2$ had similar results.

Warp specimens treated with $Mg(HCO_3)_2$ and those sprayed with MMMC do not differ significantly although the flexural rigidity value after spraying with MMMC (505 mg.cm) is about twice that of $Mg(HCO_3)_2$-treated fabric (266 mg.cm). The difference in stiffness only becomes statistically significant in test samples treated with $Mg(HCO_3)_2$ and MMMC spray when they have been cut in the weft direction. Both the weft specimens which were dipped and sprayed with MMMC are stiffened considerably, the dipping process causing the greater flexural rigidity (618 mg.cm) than spraying (153 mg.cm) (see Tables 4.6 and 4.7).

Measurements of stiffness relate directly to the fabric's hand and drape. Both hand and drape are important features of textiles which set them apart from paper and other two dimensional structures. It is no wonder, therefore, that an alteration of these qualities should bring about criticism, as has been the case with deacidificants. It is clear that polar liquids, including water, penetrate amorphous regions of cellulose and loosen and ultimately rearrange the intermolecular hydrogen bonds. Grosberg (1977) outlines some of the physical processes involved in the bending of fabrics, the deformation which the samples undergo when testing flexural rigidity. He speaks of the frictional component due to the movement of neighbouring fibres and/or yarns relative to each other which brings about a resistance to bending. Fibre slippage can be altered through the application of various finishes; that is, resistance to fibre slippage may be increased to the point of inhibiting the bending, as simply starching a fabric will do. The scanning electron microscopy photographs show evidence of some fibre to fibre adhesion, particularly when specimens are examined after a minimal amount of abrasion (see Section 4.6: Scanning Electron Microscopy and Energy Dispersive X-ray Analysis). Either an

increase in fibre friction to the point of adhesion of fibres or a rearrangement of hydrogen bonds could be at work here. It is important to note, however, that Peacock (1980), who had similar results when measuring stiffness, found that upon subsequent ageing most samples had the same degree of stiffness.

It is also relevant at this time to examine the levels of add-on. From Table 4.6, it can be seen that dipping specimens in MMMC caused the greatest weight increase and this alone may be increasing the stiffness of these samples. The dipping procedure may also allow for better saturation of the fibres than the spraying technique, which might, in turn, cause more adhesion of the fibres. If the treated fabrics are manipulated the stiffness level decreases, however, unnecessary manipulation is not recommended with fragile fabrics of historic value. It was observed that while conducting the test for flexural rigidity, the fabric became supple after only a minimal amount of handling and bending. For museum textiles which are small enough to be stored and exhibited flat, the increased stiffness may not be a problem. This stiffness will not alter the aesthetic appeal of such textiles.

4.5 Moisture Regain

Conservators and scientists who oppose the deacidification of cotton fabrics have suggested that the deacidificant on the surface of a fabric may deliquesce when it absorbs water vapour from the air. If deliquescence were to happen, the moisture absorption of the treated fabric would be greater than that of the untreated fabric. The following null hypothesis was tested:

> *f. There is no significant difference in the moisture regain of new cotton textiles treated with distilled water and the deacidifying agents, MMMC (dipped or sprayed) and $Mg(HCO_3)_2$ (dip).*

The null hypothesis was rejected. Examining Table 4.8, it is apparent that there is a significant difference in the moisture regain of specimens after treatment with water, MMMC and $Mg(HCO_3)_2$.

Table 4.8: Moisture regain (%) of cotton fabrics after treatment with distilled water and the deacidifying agents, MMMC[1] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [2,3]

Treatment			
Distilled Water	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
8.19	8.63	8.65	8.41

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'o® solution #2 for the dipping formula and solution #12 for the spraying formula).
[2]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[3]Average of 10 specimens

The two MMMC treatments, spraying and dipping, although varying in their method of application, do not differ in their effect on moisture regain. Both sets of treated fabrics have average moisture regains of approximately 8.6 %, which is not unexpected. Although the difference in moisture regain values of the deacidified fabrics and the water-treated control is statistically significant, none of the moisture regain values differs drastically from that of untreated cotton. The moisture regain for the water-treated control in this experiment was 8.19%, a value that is close to the 7 and 8 per cent moisture regain normally reported for cotton (Morton and Hearle, 1975). For the sake of comparison, mercerized cotton can be expected to have moisture regain values of up to 12 % (Morton and Hearle, 1975). The fear expressed by those who oppose the use of deacidificants is clearly unfounded as MMMC and $Mg(HCO_3)_2$ finishes only increased the moisture regain of cotton by about 0.2 - 0.4 %.

4.6 Abrasion Resistance

Both new cotton fabric and selected naturally aged cotton and linen fabrics were subjected to flexing and abrasion after treatment with MMMC (dipping or spraying) or magnesium bicarbonate. The results of abrasion testing, expressed as the average number of cycles needed to rupture the specimens, are displayed in Tables 4.9 and 4.10.

Table 4.9: Abrasion resistance (number of cycles to rupture), by the flexing and abrading method, of new cotton textiles after treatment with distilled water and the deacidifying agents, MMMC[1] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [2] [3]

Treatment			
Distilled Water	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
891±31[4]	143±6	154±6	160±9

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'o® solution #2 for the dipping formula and solution #12 for the spraying formula.
[2]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[3]Average of 20 specimens.
[4]Mean ± 95% confidence interval.

Table 4.10: Abrasion resistance (number of cycles to rupture), by the flexing and abrading method, of naturally aged cellulosic textiles after treatment with distilled water and the deacidifying agents, MMMC[1] dip, MMMC spray and $Mg(HCO_3)_2$ dip. [2] [3]

Sample Number[4]	Treatment				Null Hypothesis
	Distilled Water	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip	
3	17±1[5]	16±1	9±1	11±1	reject
13	22±3	18±2	15±2	18±2	reject
16	74±16	73±11	81±13	55±10	reject
17	27±8	33±8	23±8	23±5	accept

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'o® solution #2 for the dipping formula and solution #12 for the spraying formula.
[2]Homogeneous subsets, as determined by the Duncan's Multiple Range Test, are underlined.
[3]Average of 20 specimens.
[4]See Appendix I for description.
[5]Mean ± 95% confidence interval.

The first null hypothesis to be tested was the following:

g. There is no significant difference in the number of cycles required to rupture new cotton textiles, through flexing and abrading, among the four treatments, distilled water, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip.

The null hypothesis is rejected as there is a significant difference in the number of cycles required to rupture the treated samples as a whole and the control samples (see Table 4.9). The control specimens ruptured after an average of 891 ± 31 cycles whereas the treated specimens ruptured after approximately 140 to 170 cycles. There is no significant difference in abrasion resistance among the three types of treated fabrics.

Four naturally aged fabrics, including fabrics 3, 13, 16 and 17, were treated, then subjected to flexing and abrasion. Fabrics 3, 13, 16 and 17 are described fully in Appendix I. Briefly, however, samples 13, 16 and 17 were cotton and sample 3 was linen. Samples 3 and 13 were severely yellowed and showed signs of degradation. Samples 16 and 17 were white; sample 16 had areas of high wear and was therefore considerably weakened. These samples were chosen for abrasion testing simply because there was ample material to carry out the testing. The following null hypothesis was tested:

h. There is no significant difference in the number of flex abrasion cycles required to rupture fabric x (a naturally aged cellulosic textile) after treatment with distilled water, MMMC dip, MMMC spray or $Mg(HC)_3)_2$, where x is fabric 3, 13, 16 or 17.

The acceptance or rejection of the null hypothesis for each specimen is shown in Table 4.10. In addition, treatments which form a homogeneous group as determined by Duncan's Multiple Range Test are underlined.

Because some of the fabrics were very fragile, the number of cycles required to rupture the specimens were low; for example, specimens cut from fabric 3 and 13 required an average of 22 cycles or fewer for rupture. With the exception of fabric 13, treatment of these fragile fabrics with deacidifying agents did not make them less resistant to flexing and abrasion than the water-treated control fabric. It is difficult to determine why naturally aged textiles treated with deacidifying agents behaved differently than new cotton treated with the same agents. New cotton showed a large decrease in resistance to flex abrasion after treatment (see Table 4.9).

Abrasion manifests itself initially as surface damage and can be the result of surface-to-surface rubbing or fibre bending (Duckett, 1975). Many different types of machines are used to measure the abrasion resistance of fabrics but there seems to be little reproduceability and predictability in the test results (ASTM, 1983; Duckett, 1975). Many variables play their part in influencing the abrasion resistance of a textile such as yarn size, yarn crimp, yarn twist, crimp balance, fabric yarn count and fabric weight, so that it is difficult to make between-fabric evaluations of abrasion resistance (Galbraith, 1975). For example, each of the naturally aged fabrics and the new fabric used in abrasion testing contained different yarn and fabric geometries. In any case, it is usually appropriate to match the test method to the type of end use foreseen for a particular fabric. The flex-abrasion test method was chosen for this investigation as it subjects fabrics to both of the major sources of abrasion, bending and rubbing. The fabric is held under tension and is bent back and forth while rubbing against an abradant, in this case, a steel bar. It cannot be overly stressed, however, that this test method is very energetic and subjects the samples to forces that they would never undergo in their normal lifetime; historic textiles, properly stored, would experience a minimal amount of flexing and rubbing.

There are many possible sources of variability in the flex abrasion test procedure and it is quite likely that different operators would obtain different results when carrying out this test. When mounting the fabric sample in the testing machine, it is difficult to maintain a consistent tension (ASTM, 1983; Duckett, 1975). Variations in the abradant exist, especially due to the development of ridges of abraded textile fragments which collect at the area of abrasion. These ridges are of variable thicknesses. Despite attempts to control for fibre buildup by cleaning the area of abrasion of the accumulated fibre debris every 30 cycles, variability does occur. With these inherent problems in mind, however, it can still be observed from the results of this study that the treatment of fragile naturally aged textiles affects the abrasion resistance less than does the treatment of new cotton. In order to explain this phenomenon, the change in properties which are related to abrasion resistance must be examined.

Elongation and elastic properties are very important in the ultimate abrasion resistance of the fabric. A decrease in elongation and elasticity often results in poorer resistance to flex-abrasion (Galbraith, 1975). If the deacidifying agents coat the fibres, which judging from the SEM photography they do,(see Section 4.6; Scanning Electron Microscopy and Energy Dispersive X-ray Analysis) fibres will be immobilized. Elongation, elasticity and interfibre friction will change. It can be deduced that elongation has decreased with the application of the deacidificants because flexural rigidity or stiffness has increased (Table 4.7). Flexural rigidity varies directly as the specimen length of overhang in the cantilever test. If a fabric has low elongation and does not bend readily, the length of overhang is large as is the corresponding flexural rigidity. The water-treated fabric, with the lowest flexural rigidity (111 mg.cm) has the highest abrasion resistance (891 ± 31 cycles to rupture). Conversely, MMMC dip-treated samples had the highest flexural rigidity or stiffness (1063 mg.cm) and the lowest abrasion resistance (143 ± 6 cycles to rupture).

Due to the fragile nature of the naturally aged textiles, abrasion resistance is low (see Table 4.10). It seems likely that the fabric samples ruptured before any trend developed, making the comparison of water-treated samples and samples treated with deacidificants rather tenuous. An observation should be made, however, which seemed strikingly apparent while conducting these tests. The ridges of abraded fibres which developed when flexing and abrading new cotton fabrics were not apparent when testing the naturally aged fabrics. Thus the variability due to these ridges along the width of the sample did not occur. This could simply be because the samples broke early in the test due to their brittleness and the ridges did not have time to develop or some of the outermost fibres had already been lost during the use and ageing of the fabric. In any case, this complication in the variability at the abrasion area was not an issue for naturally aged textiles. Further abrasion studies were done in conjunction with the scanning electron microscope and will be discussed in the following section.

4.7 Scanning Electron Microscopy and Energy Dispersive X-ray Analysis

One of the main reasons for examining treated fabrics with the aid of a scanning electron microscope was to determine whether the deacidification treatments left large crystals on the surface of the fibres. It has been hypothesized that such treatments may leave sharp crystals in place which will subsequently damage fibres when textiles are handled. This damage would ultimately deny the property of reversibility to the conservator as the damage incurred to textiles by the presence of the treatments would surpass the amount of damage the textiles would have otherwise experienced. It would be impossible to restore the textiles to their original condition. The lack of reversibility would make the use of the deacidificants unacceptable to the conservator.

In this experiment, specimens were examined with a scanning electron microscope after the application of the deacidificants and after mild abrasion following the deacidification. It was hoped that the mild abrasion would simulate in-museum handling of the textiles in the longterm and allow examination of the effect of the fibres in contact with whatever crystals might be present. The scanning electron microscope would further allow inspection of the damage incurred by the crystals, if in fact, there was any.

4.7.1 Appearance of Treated Fabrics Before Abrasion

Plate 4.1 depicts typical fibres after having undergone their various treatments. The relevant treatment and the fibre's magnification are indicated below each photomicrograph. All four treatments, distilled water, MMMC dip, MMMC spray and $Mg(HCO_3)_2$ dip, are represented here.

When the control fabric, immersed only in distilled water, is examined with the aid of the scanning electron microscope (SEM) it appears much as expected (Plate 4.1a). It shows clearly the fibre convolutions and the rough fibrillar surface. It is also worth noting that debris can be found in the convolutions of cotton treated only in distilled water. This appearance and the presence of debris is typical of cotton fibres (de Gruy *et al.*, 1973; Rollins, Cannizzaro and

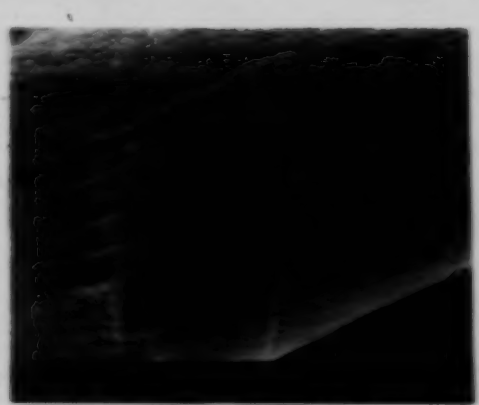
Plate 4.1 Scanning electron micrographs of new cotton after treatment

(magnified 1300X)
a. dipped in distilled water

(magnified 1300X)
b. dipped in MMMC

(magnified 1300X)
c. sprayed with MMMC

(magnified 1300X)
e. dipped in $Mg(HCO_3)_2$



(magnified 3250X)
d. dipped in $Mg(HCO_3)_2$

Goynes, 1972). The Energy Dispersive X-ray Analysis (EDAX) results (Plate 4.2), giving readings of the predominant elements present, indicate an absence of magnesium. The elements which are evident, that is, Au, Ag and Si, refer to the alloy used as the conductive coating in the sample preparation.

Examining Plate 4.1b, it is apparent that dipping the fabric in MMMC leaves the surface of the fibres very smooth. In the upper left hand corner of the photograph, one can see evidence of a coating between two adjacent fibres which appears to hold them together. Particles are very difficult to find. EDAX results confirm these observations. With or without crystals visible there is an overall presence of magnesium on the fibre surfaces (Plates 4.3 and 4.4).

The spraying of MMMC on the fabrics, as seen in Plate 4.1c, also results in few crystals and the coating is visible between the fibres. This coating appears to be quite smooth, its presence being confirmed by the EDAX results which show an overall presence of magnesium. A few crystals were analyzed with EDAX and found to contain magnesium (Appendix IV, Plates IV.1 and IV.2).

The $Mg(HCO_3)_2$ dip produces a coating on the fibres which is evident and which appears somewhat mottled. It is worth noting that this mottled surface had little effect on the flex-abrasion results when comparing the other treated new cotton fabrics with the $Mg(HCO_3)_2$ treatment (Table 4.9). There are few crystals present. The EDAX results show an overall presence of magnesium on the fibre surfaces and the few crystals present contain magnesium (Appendix IV, Plates IV.3 and IV.4). These results concur, in part, with work done by Daniels (1980) on calcium hydroxide treatment of paper. The hydroxide is converted to calcium carbonate by reaction with carbon dioxide in the air. He found that there were crystals of calcium carbonate present, but more importantly, he found that even without the crystals there was calcium present on the surface of the fibres.

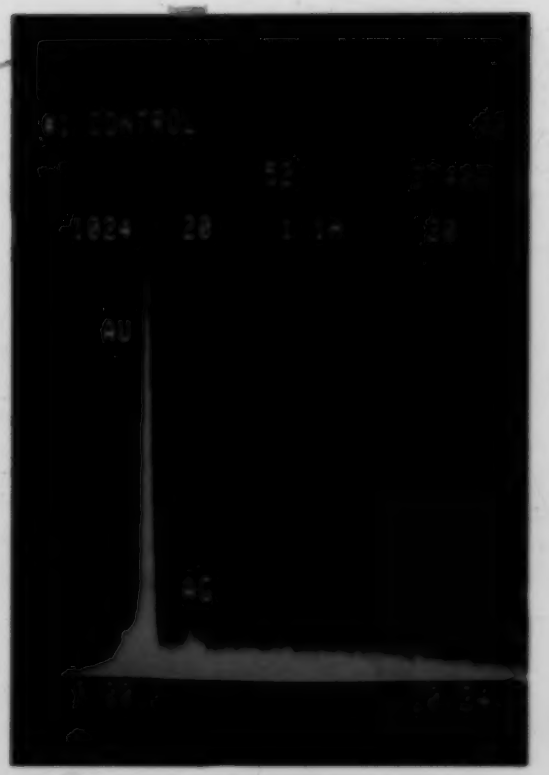


Plate 4.2 Energy dispersive X-ray analysis of control samples (treated with distilled water)

Plate 4.3 Energy dispersive X-ray analysis of an area with crystals on fabric dipped in MMMC

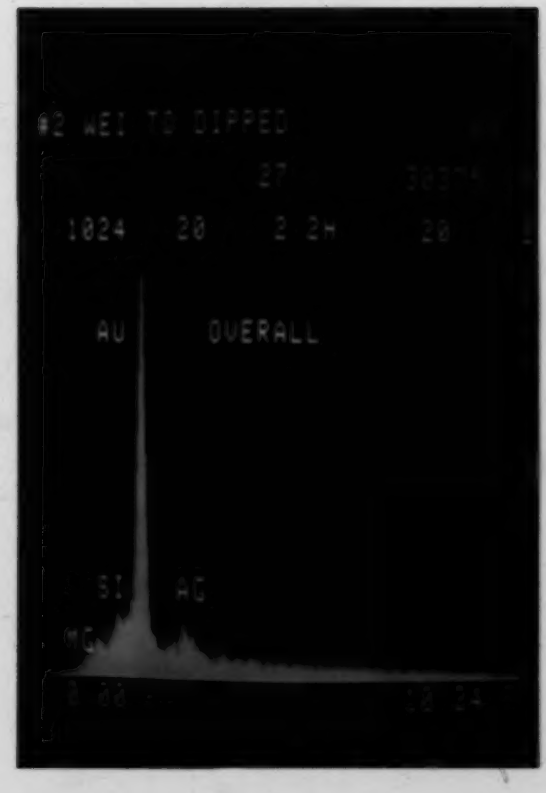


Plate 4.4 Energy dispersive X-ray analysis of an area without crystals on fabric dipped in MMMC

4.7.2 Appearance of Treated Fabrics After Abrasion

Treated fabrics were given gentle surface abrasion in the hopes of examining the steps through which the damage occurred. Abrasive wear to each specimen consisted of 1000 cycles on the Brush Pilling Tester, a machine in which a piece of cotton fabric, the untreated control, rubbed the treated fabric in a circular motion.

Microscopic examination of untreated, yet abraded, fabrics was not carried out within this study. However, studies have been done on abraded cottons in the past and this information can be used to anticipate what one might find (de Gruy *et al.*, 1973; McNally and McCord, 1960). The damage incurred by abrasion on the fibres depends to some extent on the nature and intensity of the abrasive action. Dry abrasion initially produces smoother fibre surfaces. However, it can be expected that continued friction will cause fibrillation, which is recognizable by cracks along the fibrillar structure of the cotton. Cuticle damage of the fibre surface or transverse cracking can also occur but these are much more typical signs of bending or flexing during abrasion. Ultimately one might expect to find broken ends due to the breaking of the fibres under the forces of abrasion (de Gruy *et al.*, 1973; McNally and McCord, 1960).

The photographs in Plate 4.5 are characteristic overall views of the four treated fabrics (magnification = 13X). Generally there is very little damage from the mild abrasion in all of the samples, however, it is hoped that these photographs capture the greater "hairyness" of the untreated control fabric as compared to the deacidified samples, which was evident under the microscope. This "hairyness" appears to be due to broken fibres which have been loosened and brought to the surface. There are several possible explanations as to why "hairyness" should not be as apparent on the deacidified samples as the control fabrics. Perhaps the fibres are not loosened as readily in the deacidified fabrics, as in the untreated control, or perhaps the ends break off the fabric surface of the deacidified samples more quickly than the control fabrics. The deacidification agents may provide the fabrics with a smoother surface than the untreated control fabric. This smoothness, in turn, may reduce the interfibre friction and thus reduce the number of broken ends. Perhaps the fibre ends are not loosened as readily in the deacidified

Plate 4.5 Scanning electron micrographs of new treated cotton after abrasion (magnified 13X)

a. dipped in distilled water

b. dipped in MMMC

c. sprayed with MMMC

d. dipped in $Mg(HCO_3)_2$

fabrics due to a "spot welding" of the fibres. After examining each of the photographs in Plate 4.5, little difference can be discerned among the three deacidified samples.

Plate 4.6 contains photographs of higher magnification than Plate 4.5 to allow a closer examination of each of the four treatments. As was stated at the onset of this discussion, it was hoped that through mild abrasion, one would be able to examine intermediate steps of abrasion damage, for example, fibrillation or scarring of the fibre cuticle, rather than examining the ultimate step of fibre breakage. Perhaps there would be some visual evidence of damage caused by the deacidifying coating on the fibre. Such damage was difficult to find. Either there was no damage evident or fibre breakage had already occurred. It is difficult to say what initiated the broken fibres and fibrillar cracks. They may have occurred during processing of fibres into yarns and fabric. Plate 4.6 shows examples of fibrillar damage, presumably due to the forces of abrasion, however, it must be stressed that this type of damage is not typical of the entire treated samples.

The water-treated control sample (Plate 4.6a) shows typical fibrillar damage due to abrasion. A sample dipped in MMMC (Plate 4.6b) exhibits very little damage after abrasion, however, it shows the treatment coating being pulled away from the adjacent fibre. The surface crystals have caused no noticeable amount of scarring on the fibre surfaces. Plate 4.6c is also an example of a fabric dipped in MMMC. It, too, shows the coating between the fibres. It is difficult to determine whether the fibrillar damage was caused, in part, by the adherance of the two adjacent fibres. The relative strength of inter-fibrillar bonds versus the strength of the coating layer between the two fibres is not known.

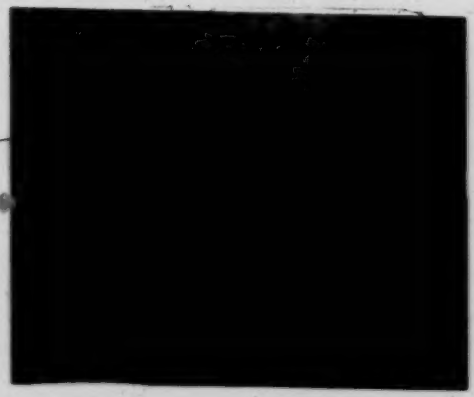
Plate 4.6d is an example of a fabric sprayed with MMMC. This photograph shows the fibre damage rather dramatically. The amount of damage that occurred in fabric production and handling, rather than during the mild abrasion carried out on the fabric during this testing, cannot be determined. However, the fibre damage seen here is not likely to be due to abrasion. There are no ragged fibriller ends, and no fibrillation. The treatment coating appears to cover the fibres. Fibre damage during processing could very definitely be a possibility. Plate 4.6d was

Plate 4.6 Scanning electron micrographs of new treated cotton after abrasion showing fibrillar damage

(magnified 1300X)
a. dipped in distilled water

(magnified 1250X)
b. dipped in MMMC

(magnified 1300X)
c. dipped in MMMC



(magnified 1000X)
d. sprayed with MMMC

(magnified 1250X)
e. sprayed with MMMC

(magnified 725X)
f. dipped in $Mg(HCO_3)_2$

included to point to the crystals present and the negligible effect they have had on the fibre surface after abrasion. Plate 4.6e is a more typical view of the sample sprayed with MMMC than the previous plate. It shows very little damage and the crystals have caused no scarring or puncturing of the adjacent fibre surface.

Plate 4.6f shows fibrillar damage on a sample dipped in $Mg(HCO_3)_2$ solution. The mottled appearance, so typical in the unabraded samples, seems to have been lost during the process of abrasion. The mottled surface layer seems to have caused no damage.

On the whole cotton fabrics after mild abrasive action may exhibit fibrillation, cuticle damage and/or broken fibre ends. It is difficult to quantify the evidence of damage and to compare the control fabrics to the deacidified fabrics, however, the deacidification samples show no more damage than the control samples. Perhaps more importantly, do the few crystals found in the unabraded, deacidified samples cause damage when abraded? No signs of cuticle damage or fibre puncturing are seen.

5. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary

It has been shown by several researchers that deacidification agents are beneficial in terms of improving the longevity of cellulosic textiles (cf. Block, 1981; Dawson, 1980; Kerr, 1982; Peacock, 1982). Most of this research has involved the accelerated ageing of fabrics after deacidification. This ageing was then typically followed by tests measuring physical or chemical changes. Such changes were the result of subsequent weakening due to oxidation and hydrolysis that the fabric had undergone during the process of accelerated ageing. Comparisons could be made regarding the effects of various deacidification agents on cellulosic textiles during ageing.

This study does not involve the degradation of cellulosic fabrics after deacidification and accelerated ageing; rather, it focuses on problems which conservators believe are caused by deacidification of textiles. Questions have been raised as to the potential problems which might be encountered if the deacidificants are present within the fibres for any extended period of time. This situation would presumably be the case if museum textiles were treated. One criterion which a textile conservator expects of any treatment applied to a textile is that the treatment be reversible, and not cause any permanent damage to the textile. Damage to the fibres has always been a concern as it has been supposed that particles are deposited within and among the fibres. In conjunction with the presence of particles there has been a concern about the change in hand and drape of the textile after treatment with the deacidificants. A change in the colour of the textiles, both dyed and aged, white textiles, has been cited as a problem with the use of these agents. Finally, the increased hygroscopic nature of the textiles has been a concern as this may cause deliquescence of the agents and a heightened reactivity of the textile surface.

The effect of three deacidifying agents on selected properties of cellulosic fabrics was examined. A variety of cellulosic textiles was used: new cotton, cotton aged rapidly by irradiation with a 50 or 75 Mrad dose of gamma radiation, naturally aged cotton and linen and

new cotton fabric dyed in a range of seven colours. The deacidifying agents included methoxy magnesium methyl carbonate (MMMC) which is a commercial product (Wei T'oo) applied from a fluorocarbon/alcohol solvent. Fabrics were dipped in Wei T'oo solution #2 or sprayed with Wei T'oo solution #12. The third agent was an aqueous solution of magnesium bicarbonate in which samples were dipped. Table 5.1 summarizes the results of physical tests on new cotton which was deacidified.

The scanning electron micrographs of treated fibres are surprising as they show that few particles are present on the fibres after they have been deacidified with MMMC or $Mg(HCO_3)_2$. It is also relevant to note that the control samples, treated only with distilled water, contain as many foreign particles as do the deacidified specimens. Furthermore, energy dispersive X-ray analysis of the surface of the fibres, regardless of which deacidifying agent was in place, reveals the presence of magnesium eventhough few crystals are seen. Those particles which are present are found to contain magnesium. The fibre surface of the MMMC-treated fabrics, whether dipped or sprayed, clearly display the presence of a smooth coating that is often most evident between adjacent fibres. Photomicrographs suggest that the deacidifying agent may be holding adjacent fibres together. The finish produced by the $Mg(HCO_3)_2$-dipped fabrics has a mottled appearance. This mottled surface does not appear to have an adverse effect on the textiles when examining the flex-abrasion results. When deacidified fabrics are gently abraded they show no more evidence of damage than do fabrics treated only with distilled water, under the same conditions. Although difficult to quantify, it seems that the presence of the deacidificants does not increase the severity of the abrasion damage. Also, there is no evidence that the crystals present cause surface fibre damage or puncturing of adjacent fibres during the mild abrasive action.

The moisture regain of deacidified fabrics showed a slight increase (0.2 - 0.4 %) over the moisture regain of the control samples (Table 5.1). With such a slight increase, it must be assumed that the deacidified fibres are not particularly hygroscopic as feared by some conservators.

Table 5.1: Weight increase (% add-on), stiffness (relative to control), moisture regain (%), flex-abrasion (% of control) and colour change (GSFS)[1] of new, undyed cotton samples after treatment with distilled water or the deacidifying agents, MMMC[2] dip, MMMC spray or $Mg(HCO_3)_2$ dip.[3]

Test Parameter[4]	Treatment			
	Distilled water	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
weight increase	-	4.76	2.01	3.89
stiffness	1	9.6	2.5	1.4
moisture regain	8.2	8.6	8.6	8.4
flex abrasion	100	16	17	18
colour change[5]	-	4.5	4.8	4.2[6]

[1]Gray Scale For Staining
[2]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula).
[3]Results from other experiments, specifically, the colour change of naturally aged cellulosic textiles, the colour change of cottons aged by accelerated means, the colour change of cottons commercially dyed, upon treatment can be found within Chapter 4.
[4]For each procedure 10 specimens were tested, except for flex abrasion in which 20 specimens were tested.
[5]Average of 3 evaluations.
[6]The expert panel described colour change as being whiter.

There is an increase in the flexural rigidity or stiffness of fabrics which are treated with the deacidifying agents, however, the increase depends on the agent and its method of application. Dipping fabrics in MMMC causes about a 10 fold increase in stiffness, whereas little change was observed when fabrics were dipped in the aqueous-based $Mg(HCO_3)_2$ treatment. Dipping in MMMC causes a greater weight gain (4.76 %) than spraying with MMMC (2.01 %) or dipping with $Mg(HCO_3)_2$ (3.89 %). This increase in weight alone may be a possible explanation for the increase in flexural rigidity of the deacidified samples. It is hoped

that with further experimentation a better method of application than dipping in MMMC might be found.

The results of tests for resistance to flexing and abrasion are surprising, judging from the appearance of treated fibres under the electron microscope. New cotton, treated with each of the deacidifying agents, ruptured after approximately 16-18 % of the number of cycles required to rupture new cotton treated with distilled water. The decrease in the resistance to abrasion may be related to a change in inter-fibre friction or an increase in flexural rigidity and fabric weight due to the presence of the deacidifying agents. In any case, it is cause for concern. This decrease in abrasion resistance found in new cottons is not as great a problem in naturally aged cellulosic fabrics. In Table 5.2 it can be seen that the number of cycles to rupture the fabrics varied from 53 to 122 % of the water treated control fabric. The fragile nature of the naturally aged fabrics may be the underlying reason that they had similar resistance to abrasion before and after treatment. Perhaps rupture occurred early in the testing, before the effects of the deacidifying agents could be felt, thus offsetting the actual effect of the presence of the agents. In this way, no trend developed in the testing procedure which would be useful in predicting the effects of a deacidificant on naturally aged fabrics.

Fears that the deacidificants will change the colour of already dyed fabrics are lessened with this study. None of the agents causes a serious change in the colour of the dyed fabrics. It must be stressed, however, that the cotton fabrics examined were dyed commercially with modern dyestuffs. Ancient fabrics containing natural dyes may be more sensitive to the alkalinity of the deacidificants. Fabrics printed with pigment colours are of particular concern as pigment colours on paper were found to have been affected in a study done by Daniels (1982).

New white cotton fabrics treated with MMMC and $Mg(HCO_3)_2$ exhibit minimal colour change; in fact, $Mg(HCO_3)_2$ whitens the specimens slightly. When naturally aged cellulosic textiles are treated, the colour change is greater than when new cotton is treated. Nevertheless, the darkening of the specimens can only be described as a slight change. Problems arise when

Table 5.2: Summary of flex abrasion results (% of control) of new, undyed cotton and naturally aged cellulosic textiles after treatment with distilled water or the deacidifying agents, MMMC[1] dip, MMMC spray and $Mg(HCO_3)_2$ dip.

Description	Sample Number[4]	Treatment			
		Distilled water	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
new cotton	-	100	16	17	18
aged linen	3	100	94	53	65
aged cotton	13	100	82	68	82
aged cotton	16	100	99	109	74
aged cotton	17	100	122	85	85

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula).
[3]Average of 20 specimens.
[5]Cycles to rupture was used as the end point of each test.
[4]Refer to Appendix 1 for a description of each sample.

samples aged by accelerated means (γ-irradiation) are deacidified because the specimens contain a considerable amount of water soluble degradation products. These products cause a yellowing of the treatment bath and they tend to migrate across the fabric to its edges, resulting in an uneven colour of the artefact after treatment. It can be concluded that whenever possible fabrics which have aged considerably should be washed before treatment with deacidification agents in order to avoid this migration of the water soluble, yellow degradation products. On the whole, therefore, the application of the treatments has only a minimal effect on the colour of the samples.

5.2 Conclusions

The three deacidifying agents examined in this study, methoxy magnesium methyl carbonate (MMMC) in the dipping and spraying formulae, and magnesium bicarbonate ($Mg(HCO_3)_2$), were selected because they are commonly used by paper conservators and previous research indicated that they could prolong the life of cotton fabric subjected to accelerated ageing (Kerr, 1982). The nature of an artefact may predetermine which agent is the most suitable for use. It is, however, important to know beforehand the type and extent of changes one can expect with each deacidification agent.

The MMMC formulae are applied from a nonaqueous (fluorocarbon) solvent and, as such, are suitable for textiles which are too fragile for immersion in water or contain embellishments, designs or colours which are adversely affected by water. The MMMC spraying formula is particularly suitable for an artefact that is too large or cumbersome because of its three-dimensionality to immerse into a solution.

The $Mg(HCO_3)_2$ formula is an aqueous-based deacidification agent, appropriate for a textile which can withstand immersion in a water bath. Colours must initially be tested to confirm their stability in water. The inherent structure of the artefact must also allow a water bath. If all of the criteria are met, then $Mg(HCO_3)_2$ would indeed be a good choice as a deacidification agent. Table 5.3 can be used as a guide in selecting the appropriate treatment as the effect of each deacidification agent on the samples tested is outlined.

The data on colour change reflects a "considerable to noticeable change" when γ-irradiated cotton is treated with deacidification agents. It is presumed, however, that the application of the aqueous $Mg(HCO_3)_2$ would have less effect on these cellulosic textiles, which contain a considerable amount of the water soluble degradative products, if they are washed beforehand. If this treatment has been selected because it is suitable for a particular artefact, then likely, the textile can withstand washing in water. Therefore, the problem of increased colour change can, in all probability, be minimized in the case of the $Mg(HCO_3)_2$ treatment.

Table 5.3: Summary of the effects of MMMC[1] dip or spray and $Mg(HCO_3)_2$ dip on the physical properties of various cellulosic fabrics.[2]

Test Parameter	Fabric Type	Treatment		
		MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
Colour difference	new, white cotton	nil	nil	slight whitening
	new, dyed cotton	nil	nil	nil
	cotton aged by γ-irradiation	noticeable	noticeable	considerable
	naturally aged cellulosic	slight to noticeable	negligible to noticeable	negligible to slight
Stiffness		considerable	slight	negligible
Moisture regain		slight	slight	slight
Flex abrasion	new cotton	considerable	considerable	considerable
	naturally aged cellulosics	nil to negligible	nil to negligible	nil to negligible

[1]MMMC is methoxy magnesium methyl carbonate (commercially available as Wei T'oo solution #2 for the dipping formula and solution #12 for the spraying formula).
[2]Descriptive terms for greatest change to least change are ranked as follows: considerable > noticeable > slight > negligible > nil.

Fabrics which are not to be washed might be considered for treatment with MMMC. Again examining the data on colour change of γ-irradiated cotton the results show a noticeable change after treatment with MMMC. Studies have shown that colour change brought about with the application of the deacidifying agents is, however, offset with subsequent ageing (Kerr, 1982; Peacock, 1983). For many textiles the increase in stiffness due to the presence of the MMMC treatment, particularly the dipping formula, may be undesireable and may preclude

the use of the dipping formula. For those textiles that are small, and can be stored and exhibited flat, the alteration in hand and drape may not affect them aesthetically and, therefore, this physical change may not be a problem. The initial increase in stiffness which becomes less noticeable when the textile has been handled for a short time, may be more than offset by the potential increase in lifetime of the textile.

The attributes of an artefact more or less prescribe the type of deacidification treatment which is the most suitable. Therefore, the underlying question is not so much which agent to use, but whether or not to use a deacidification agent at all. There is no doubt however that, through the use of these products, the pH level of the textile is raised to an alkaline level and the longevity of the textile is increased (Kerr, 1982). More experimentation is required to find techniques for applying these agents. Perhaps there are ways to minimize the stiffness and maximize the resistance to flexing and abrading. Nevertheless, with the results of this study in hand, one is better able to make an informed decision as to which product to use and the type of changes one might expect with its use.

5.3 Recommendations

5.3.1 Recommendations for Future Study

As a consequence of this study, interesting questions have surfaced which warrant some examination. There is a definite problem associated with the application of deacidifying agents, particularly methoxy magnesium methyl carbonate (MMMC) in the dipping formula. It is important to devise a method of application that will not increase the stiffness of the fabric.

The degree of resistance to flexing and abrasion exhibited by a textile is related to its stiffness. It is unfortunate, therefore, that due to the lack of sufficient fabric tests for flexural rigidity before and after deacidification were not carried out on the naturally aged cellulosic textiles in this study. Such tests might have helped to explain the ambiguous results obtained in testing the resistance to flexing and abrasion of new cottons and naturally aged cottons. The

presence of the deacidificants had a profound effect on the new fabric and a negligible effect on the naturally aged. Therefore, in order to explain these results more clearly, tests for stiffness of the naturally aged textiles, studied in the same context, should be carried out.

When naturally aged cellulosic textiles were first soaked in water and the pH of the water extracts measured (Appendix I) the soak water was not very acidic. There could be several reasons for the lack of acidity. The textiles may not have been damaged by oxidation, which results in the formation of carboxylic acid groups (and water soluble degradation products) and hence lowers the pH level. The textiles may have been washed prior to their being obtained for testing. Useful information could be provided by testing the colour change caused by deacidification when samples which initially have a lower pH level than those listed in Appendix I are treated. If highly oxidized cellulosics were deacidfied, it is possible that they would exhibit a colour change comparable to that observed in the γ-irradiated samples.

Scanning electron microscopy could be used to answer a specific question. It is important to know if there are more crystals along the line of evaporation, created during the drying stage of the treatment, than are found elsewhere on the textile. This area could potentially be a region of increased susceptibility to damage, hence contributing to the irreversibility of the deacidification treatments.

The surface of naturally aged fibres differs from fibres aged by accelerated means. Berry *et al.* (1977) found the surfaces of naturally aged fibres (taken from ancient Peruvian grave textiles) to be more granular than those aged through various accelerated means. They supposed that the environment in which the natural ageing took place, that is, the dust in which they were found, contributed to this granularity. Nevertheless it is important to examine, with the aid of the scanning electron microscope, naturally aged fibres which have been deacidified.

Museum personnel frequently criticize research based on samples aged by accelerated means because the accelerated ageing conditions vary considerably from conditions that artefacts encounter during normal ageing. Naturally aged specimens should be treated with deacidification agents and allowed to age further by natural means, if possible. Such an

experiment would address a question raised by several conservators: will deacidifying agents help to slow down the degradation of old fabrics which are treated? Studies along these lines may help in understanding the need for deacidification. Block (1982) questions the effectiveness of deacidificants on naturally aged textiles. He suggests that with time the fibre's crystallinity increases and, therefore, the number of accessible hydroxyls which can form carboxyl groups will decrease. In addition, he believes that the carboxyl groups which cause degradation decay away, thus, the alkaline treatment intended for the carboxyl groups is not necessary for aged textiles, although it seems quite effective for new cellulosic textiles. A complete study dealing with the deacidification and subsequent ageing of already aged samples is therefore in order.

5.3.2 Recommendations for the Preparation and Application of the Deacidification Agents

MMMC (dipping formula): This product is commercially available from Wei T'o Associates, Incorporated. Relative humidity plays an important part in the successfulness of a treatment. For this reason several formulations containing varying amounts of methanol or ethanol are available which work effectively at different levels of relative humidity. The higher the alcohol content in the fluorocarbon/alcohol solvent, the less likelihood there is for precipitation of MMMC when samples containing moisture are dipped. For this reason, solution #2, which contains methanol, was chosen as being the most suitable for dipping specimens.

The instructions enclosed with the Wei T'o® treatment are intended for use on paper. Textiles can be handled similarly, however, the time allotment for the treatment is much more crucial with textiles than with paper. An increase in stiffness, which appears to be directly related to the length of time the artefact remains in the bath, is not as vital to paper, which is usually two-dimensional and already displays some rigidity, as it is to textiles. Textiles are required to retain flexibility, softness and handle much as they did originally. These are tactile and aesthetic qualities which should be changed only minimally, if at all, after treatment.

Therefore, it is important that the dipping time be kept to a minimum; 10 seconds was used in this study as longer immersion times increased the add-on and the propensity for precipitation to occur.

The solution should be used directly and quickly. It should be deep enough to allow free immersion of the textile into the solution. Precipitates do accumulate on the textile if sufficient solution is not used and/or the solution has been exposed to the open air for longer than 15 to 20 minutes. The precipitates are white and cause a visible colour change to the fabric being treated. For these reasons, too, used solutions or milky solutions should be discarded.

Keeping the minimal time span of the treatments in mind, the supplies and the artefacts should be as organized as possible before treatment. Provisions should be made for easy lifting of the artefact in and out of the treatment bath. In this way, the artefact will undergo the least amount of stress.

The drying of the textile should be done on a flat surface. In this study a glass surface was used, but some of the deacidificant adhered to the surface when the textile was removed after drying. Perhaps the drying might be best accomplished on a screen to leave the maximum amount of the deacidification agent on the textile.

MMMC (spraying solution): For this study Wei T'oo solution #12 was used as it is more resistant to precipitation caused by moisture than is solution #10 or #11 since ethanol is the primary solvent rather than trichlorotrifluoroethane. In solutions #10 or #11 the reverse is true, that is, trichlorotrifluoroethane is the primary solvent and alcohol, whether methanol or ethanol, is the secondary solvent. The appropriate formulation can be selected for a particular RH characteristic of the environment in which it will be used. The suggestions for application outlined in "*Hints for Better Spraying*" (Smith, Note 2), distributed with the product, should be followed. With careful back and forth motion of the spray can, holding it at a consistant distance of 13 - 18 cm (5 - 7 in) an allover coverage of the deacidifying agent will be insured. It is recommended that the textile be supported in the vertical position for spraying as this will eliminate chances of the solution dripping from the nozzle. Also a finer, more even coverage

will be achieved in the vertical position than in a slanted or horizontal position. With textiles of a fragile nature, or textiles which are very large, the vertical position may not be possible. A slightly angled position or the horizontal position must then be considered.

$Mg(HCO_3)_2$ (dipping solution): This solution can be mixed by the conservator using few supplies. The pamphlet, entitle "*Preparation of Solutions of Magnesium Bicarbonate for Deacidification of Documents*" (Wilson *et al.*, 1979), is a useful resource if this treatment is to be considered.

The ease of preparation and the time required depends on the particle size of the powder used, whether $Mg(OH)_2$ or $MgCO_3$, and the purity of the CO_2 used (Wilson *et al.*, 1978). Also, with the use of dry ice as the source of CO_2, the rate of CO_2 generation may vary. Therefore there is little consistency in the length of time required to achieve a solution. Often undissolved particles remain, which also take a varying length of time to settle to the bottom. It is recommended that the clear solution be decanted in order that the conservator will be able to recognize if precipitation is occurring, which in turn, might indicate a decrease in the concentration of the $Mg(HCO_3)_2$ solution. The instructions of Wilson *et al.* (1979) give an indication of the time involved in making up $Mg(HCO_3)_2$. It is recommended that the solution be used within one or two days after preparation as there is little information regarding the change of strength with time (Wilson *et al.*, 1978).

It is suggested that whenever possible textiles which have become yellow with age be washed before treatment. This procedure would remove much of the water soluble yellow degradation products and prevent their migration to the fabric edges as was seen in this study when yellowed γ-irradiated samples were deacidified. Hey (1979) suggests that, through washing before treating, the deacidification agent is used to its maximum potential. A residual alkaline reserve is left in place rather than the alkalinity being forfeited immediately upon treatment, through the neutralization of the acidic water soluble degradation products.

Hey (1979) also suggests that the drying stage between washing the textiles and treating them be omitted. Wetting out with water swells the cellulosic fibres. Treatment of a fabric in

the swollen state should enhance the absorption of the deacidification agents into the amorphous regions of the fibres. This practice was not carried out within this study, but might be worthy of consideration.

The treatment bath should be sufficient to allow easy immersion of the textiles. The length of time of immersion for this study was 0.5 hours and was found to be long enough for adequate add-on. After treatment the textiles should be allowed to dry on a flat surface, perhaps a screen which would allow for a maximum amount of deacidificant to stay on the textile and facilitate rapid drying of the fabric.

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APPENDIX I

DESCRIPTION OF NATURALLY AGED CELLULOSIC TEXTILES

Table I.1: Information pertaining to cellulosic fabrics aged naturally.

Sample Number[1]	Description	Condition	pH[2]	Source
1	linen tea cloth	-white, in excellent condition	6.9	Marian Centre[3]
2	fine linen tea cloth	-a grey white with a number of holes	6.7	Marian Centre
3	large linen runner	-very yellow to tan in colour, holes indicative of degradation	6.7	Marian Centre
4	cotton napkins	-white, a few yellow stains; good condition	6.5	Marian Centre
9	white cotton shirt, about 1960	-very yellowed, with varying degrees of yellowness; otherwise in good condition	5.4	N. Kerr
11	lightweight cotton shirt	-very white, heavily starched, a few holes due to construction methods rather than degradation	6.9	U. of A. (HCC)[4]
12	cotton fragment, very light weight	-yellowed, stained; some signs of degradation	5.8	U. of A. (HCC)
13	cotton nightgown, about 1900's	-yellowed; good condition	5.5	U. of A. (HCC)
16	cotton sheet, about 1960	-white, samples selected from regions of low wear	-	E. Bitner
17	cotton sheet, about 1960	-white, samples selected from regions of high wear; very worn, fragile with many holes	-	E. Bitner

[1] For many pieces the date of origin was difficult to ascertain.
[2] pH of initial water bath. Water/sample ratio of 70:1 by weight to approximate the proportions used for aqueous extract pH's of samples.
[3] Gift store of the Order of the Sisters of Cumberland, Edmonton, Alberta.
[4] University of Alberta Historic Costume and Textile Study Collection.

APPENDIX II

PRELIMINARY RESULTS

Table II.1: Weight increase (% add-on) and pH of new cotton fabric after treatment with deacidifying agents.

Description of Treatment	% Add-on[1]	pH[2]	
		aqueous extract	surface electrode[3]
Water	-	6.9	6.5
MMMC dip[4]			
10 s	1.2	10.5	10.3
60 s	3.9		
120 s	4.6		
240 s	6.5		
300 s	7.3	10.6	9.7
$Mg(HCO_3)_2$ #1			
0.5 h dip	0.08	10.3	9.9[5]
$Mg(HCO_3)_2$ #2[6]			
2 x 0.5 h dip	0.02	10.8	10.0

[1]Average weight increase of 2 samples.
[2]Average of 4 readings
[3]Surface electrode readings were taken as well as aqueous extract readings as it is recognized that in many cases this nondestructive test is the only one appropriate for historic artifacts. For the purpose of identifying the relative alkalinity or acidity of a textile it can be seen from the above readings that surface electrode readings are adequate.
[4]MMMC is methoxy magnesium methyl carbonate in this case the commercial product Wei T'oo dipping solution #2.
[5]Great variability in the readings (range 9.2 to 10.4).
[6]Penetration of liquid during the second dip was poor as the fabric seemed impervious to the liquid and had to be turned and agitated somewhat. Lower % add-on may be due to loss of original $Mg(CO_3)_2$ deposit or may reflect variability in % add-on.

APPENDIX III

STATISTICAL ANALYSES

Table III.1: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, new, undyed cotton.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	9.1306	4.5653	90.9447	.0000
WITHIN GROUPS	27	1.3554	.0502		
TOTAL	29	10.4860			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3
MEAN	0.6800

SUBSET 2

GROUP	GRP 2
MEAN	1.1830

SUBSET 3

GROUP	GRP 4
MEAN	2.1830

Table III.2: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #1 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	4.6383	2.4142	173.9885	.0000
WITHIN GROUPS	16	.2092	.0139		
TOTAL	17	5.0376			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	0.3667

SUBSET 2

GROUP	GRP 2
MEAN	0.9667

SUBSET 3

GROUP	GRP 3
MEAN	1.5150

Table III.3: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #2
(number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	10 2064	5 1032	67 0868	0000
WITHIN GROUPS	15	1 1411	0761		
TOTAL	17	11 3495			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	0 4187

SUBSET 2

GROUP	GRP 2	GRP 3
MEAN	1 8700	2 0550

Table III.4: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #3
(number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	2 9567	1 4784	1 8978	1842
WITHIN GROUPS	15	11 6920	7795		
TOTAL	17	14 6507			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4	GRP 3	GRP 2
MEAN	1 2983	1 5283	2 2500

Table III.5: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #4
(number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	3.8793	1.4398	3.9372	.0309
WITHIN GROUPS	15	7.3297	.4820		
TOTAL	17	10.1992			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 2	GRP 3	GRP 4
MEAN	0.6433	1.6833	1.7000

Table III.6: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #9
(number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	8.3187	4.1593	3.0780	.0760
WITHIN GROUPS	15	20.2891	1.3526		
TOTAL	17	28.6078			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4	GRP 2
MEAN	1.1633	2.1233

SUBSET 2

GROUP	GRP 2	GRP 3
MEAN	2.1233	2.8217

Table III.7: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #11 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	.7868	.3863	38.7978	.0000
WITHIN GROUPS	16	.1024	.0106		
TOTAL	17	.8890			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	0.4883

SUBSET 2

GROUP	GRP 3	GRP 2
MEAN	0.8750	0.8767

Table III.8: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #12 (number corresponds to sample number in Appendix I)

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	3.5718	1.8359	3.4487	.0565
WITHIN GROUPS	15	7.9824	.5322		
TOTAL	17	11.5541			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4	GRP 2
MEAN	1.2450	3.1233

SUBSET 2

GROUP	GRP 3	GRP 3
MEAN	3.1233	3.2667

Table III.9: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #13
(number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	3	12.8217	5.4109	27.9981	.0000
WITHIN GROUPS	16	3.4346	.2290		
TOTAL	17	16.2563			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	0.6533

SUBSET 2

GROUP	GRP 3
MEAN	2.1383

SUBSET 3

GROUP	GRP 2
MEAN	2.6983

Table III.10: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #16
(number corresponds to sample number in Appendix I)

ANALYSIS OF VARIANCE

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	2	6.6686	3.3343	26.6058	.0000
WITHIN GROUPS	15	1.8148	.1277		
TOTAL	17	8.4834			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	0.6533

SUBSET 2

GROUP	GRP 2	GRP 3
MEAN	1.6550	2.0550

Table III.11: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated, naturally aged cellulosic textile #17 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	14.0467	7.0234	165.8711	.0000
WITHIN GROUPS	15	.6359	.0424		
TOTAL	17	14.6826			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE.

SUBSET 1

GROUP	GRP 4
MEAN	0.8133

SUBSET 2

GROUP	GRP 3
MEAN	2.1483

SUBSET 3

GROUP	GRP 2
MEAN	2.9583

Table III.12: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated cotton, γ-irradiated with a dosage of 50 Mrad.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	31.6244	15.8122	3.7884	.0465
WITHIN GROUPS	15	62.5840	4.1723		
TOTAL	17	94.2084			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE.

SUBSET 1

GROUP	GRP 3	GRP 2
MEAN	5.3533	5.8567

SUBSET 2

GROUP	GRP 4
MEAN	6.2650

Table III.13: Oneway analysis of variance and Duncan's Multiple Range Test for colour change of treated cotton, γ-irradiated with a dosage of 75 Mrad.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	28.8727	14.2863	9.9652	.0018
WITHIN GROUPS	15	21.5471	1.4365		
TOTAL	17	50.1208			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 2
MEAN	5.5600	6.4333

SUBSET 2

GROUP	GRP 4
MEAN	8.8467

Table III.14: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed yellow.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	10.4770	5.2385	114.0188	.0000
WITHIN GROUPS	27	1.2405	.0459		
TOTAL	29	11.7175			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	1.0640

SUBSET 2

GROUP	GRP 2	GRP 3
MEAN	2.3860	2.3470

Table III.15: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed green.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	.649[illegible]	.3249	3.3143	.0560
WITHIN GROUPS	27	1.686[illegible]	.0980		
TOTAL	39	2.335[illegible]			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 2
MEAN	0.6950	0.7000

SUBSET 2

GROUP	GRP 4
MEAN	0.9570

Table III.16: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed mauve.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	.7535	.3784	3.8495	.0048
WITHIN GROUPS	27	1.5656	.0580		
TOTAL	39	2.3251			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 4
MEAN	0.7950	0.8200

SUBSET 2

GROUP	GRP 2
MEAN	1.1400

Table III.17: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed coral.

ANALYSIS OF VARIANCE

SOURCE	D.F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	.1056	.0578	2.2578	.1206
WITHIN GROUPS	27	1.1504	.0426		
TOTAL	29	1.3561			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 2	GRP 4
MEAN	0.7180	0.8300	0.8130

Table III.18: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed navy blue.

ANALYSIS OF VARIANCE

SOURCE	D.F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	2	.0285	.0143	.1000	.8682
WITHIN GROUPS	27	2.3976	.0888		
TOTAL	29	2.4276			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4	GRP 3	GRP 2
MEAN	0.6770	0.7030	0.7530

Table III.19: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed light blue.

ANALYSIS OF VARIANCE

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	2	.8613	.4307	7.9818	.0019
WITHIN GROUPS	27	1.4266	.0539		
TOTAL	29	2.2879			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE.

SUBSET 1

GROUP	GRP 3
MEAN	.0.3900

SUBSET 2

GROUP	GRP 2	GRP 4
MEAN	0.8450	0.7880

Table III.20: Oneway analysis of variance and Duncan's Multiple Range Test for colour change after treatment of cotton commercially dyed aqua.

ANALYSIS OF VARIANCE

SOURCE	D.F.	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB.
BETWEEN GROUPS	2	2.1406	1.0703	45.8853	.0000
WITHIN GROUPS	27	.6302	.0233		
TOTAL	29	2.7706			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE.

SUBSET 1

GROUP	GRP 3
MEAN	0.8710

SUBSET 2

GROUP	GRP 4
MEAN	0.7330

SUBSET 3

GROUP	GRP 2
MEAN	1.3010

Table III.21: Oneway analysis of variance and Duncan's Multiple Range Test for flexural rigidity, in the warp direction, of treated, new cotton.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	17518912.24	5839637.416	75.4987	.0000
WITHIN GROUPS	36	2784168.585	77338.3496		
TOTAL	39	20303080.83			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 1	GRP 4
MEAN	195.3043	366.3750

SUBSET 2

GROUP	GRP 4	GRP 3
MEAN	366.3750	604.5840

SUBSET 3

GROUP	GRP 2
MEAN	1027.6356

Table III.22: Oneway analysis of variance and Duncan's Multiple Range Test for flexural rigidity, in the weft direction, of treated, new cotton.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	2038675.830	679558.6400	355.4581	.0000
WITHIN GROUPS	36	68824.1812	1911.7828		
TOTAL	39	2107500.101			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 1	GRP 4
MEAN	82.6626	90.1804

SUBSET 2

GROUP	GRP 3
MEAN	152.6807

SUBSET 3

GROUP	GRP 2
MEAN	517.6667

Table III.23: Oneway analysis of variance and Duncan's Multiple Range Test for moisture regain of treated, new cotton.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	1.3879	.4626	72.3766	.0000
WITHIN GROUPS	36	.2301	.0064		
TOTAL	39	1.6180			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 1
MEAN	6.1840

SUBSET 2

GROUP	GRP 4
MEAN	6.4080

SUBSET 3

GROUP	GRP 2	GRP 3
MEAN	6.6280	6.6530

Table III.24: Oneway analysis of variance and Duncan's Multiple Range Test for abrasion resistance of treated, new cotton.

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	819503.637	273210.546	2182.2883	.0000
WITHIN GROUPS	76	9510.2500	125.1349		
TOTAL	79	829113.887			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 2	GRP 4	GRP 3
MEAN	142.9000	163.8000	180.3500

SUBSET 2

GROUP	GRP 1
MEAN	881.4000

Table III.25: Oneway analysis of variance and Duncan's Multiple Range Test for the abrasion resistance of treated, naturally aged cellulosic textile #3 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	845.6375	281.8792	38.9288	.0000
WITHIN GROUPS	76	555.0000	7.3184		
TOTAL	79	1401.6375			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 4
MEAN	9.4500	11.0800

SUBSET 2

GROUP	GRP 2	GRP 1
MEAN	16.3000	17.0800

Table III.26: Oneway analysis of variance and Duncan's Multiple Range Test for the abrasion resistance of treated, naturally aged cellulosic textile #13 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	530.5500	176.8500	7.1760	.0003
WITHIN GROUPS	76	1873.0000	24.6447		
TOTAL	79	2403.5500			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 3	GRP 2	GRP 4
MEAN	15.3500	17.7500	17.7500

SUBSET 2

GROUP	GRP 1
MEAN	22.4500

Table III.27: Oneway analysis of variance and Duncan's Multiple Range Test for the abrasion resistance of treated, naturally aged cellulosic textile #16 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	7503.9375	2834.6458	3.8388	.0165
WITHIN GROUPS	76	54689.4500	719.5822		
TOTAL	79	62193.3875			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4
MEAN	64.7500

SUBSET 2

GROUP	GRP 2	GRP 1	GRP 3
MEAN	72.8500	73.7000	81.3500

Table III.28: Oneway analysis of variance and Duncan's Multiple Range Test for the abrasion resistance of treated, naturally aged cellulosic textile #17 (number corresponds to sample number in Appendix I).

ANALYSIS OF VARIANCE

SOURCE	D F	SUM OF SQUARES	MEAN SQUARES	F RATIO	F PROB
BETWEEN GROUPS	3	1081.5375	383.8458	1.3404	.2676
WITHIN GROUPS	76	20639.4500	271.4533		
TOTAL	79	21721.9875			

HOMOGENEOUS SUBSETS (SUBSETS OF GROUPS, WHOSE HIGHEST AND LOWEST MEANS DO NOT DIFFER BY MORE THAN THE SHORTEST SIGNIFICANT RANGE FOR A SUBSET OF THAT SIZE)

SUBSET 1

GROUP	GRP 4	GRP 1	GRP 3	GRP 2
MEAN	23.2000	28.8500	28.3500	33.4500

APPENDIX IV

ENERGY DISPERSIVE X-RAY ANALYSIS

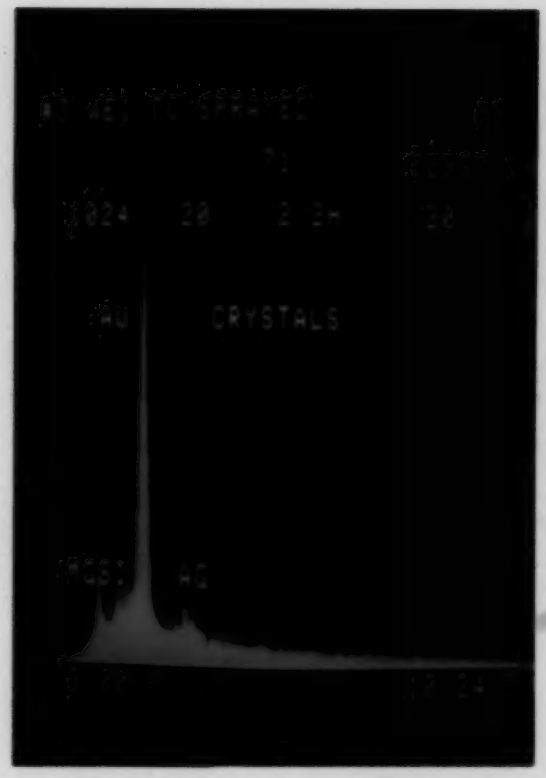

Plate IV.1 Energy dispersive X-ray analysis of an area with crystals on fabric sprayed with MMMC

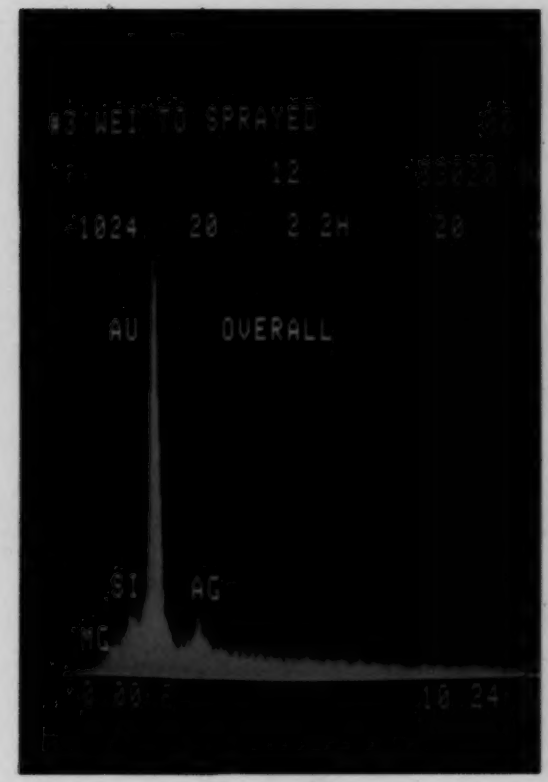

Plate IV.2 Energy dispersive X-ray analysis of an area without crystals on fabric sprayed with MMMC

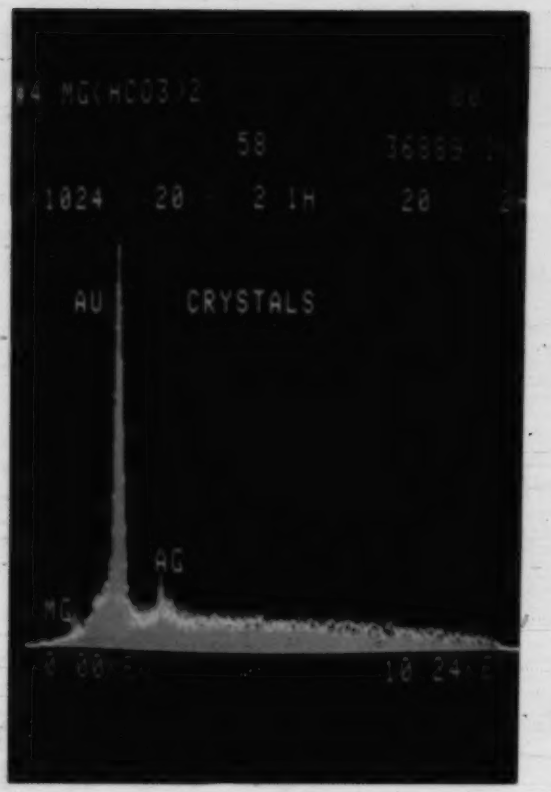

Plate IV.3 Energy dispersive X-ray analysis of an area with crystals on fabric dipped in $Mg(HCO_3)_2$

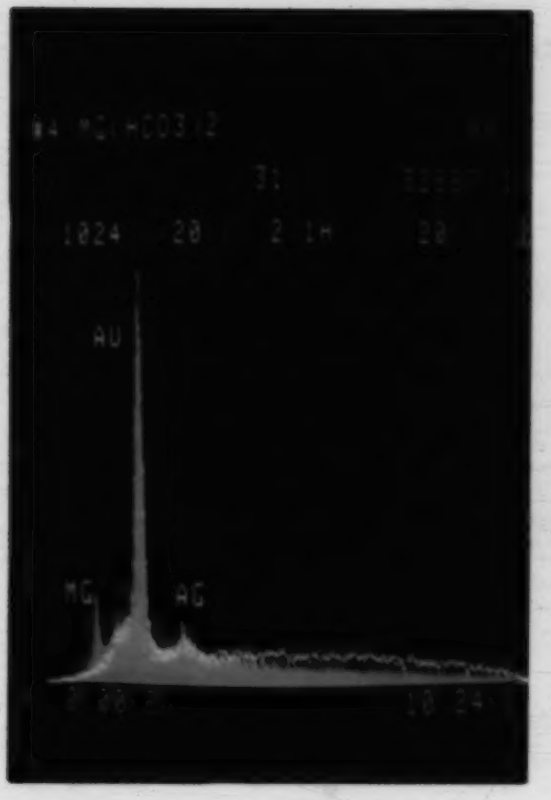

Plate IV.4 Energy dispersive X-ray analysis of an area without crystals on fabric dipped in $Mg(HCO_3)_2$

APPENDIX V

RAW DATA

Table V.1: Colour difference readings (CIELAB units)[1] of new white cotton textiles upon treatment with the deacidifying agents, MMMC[2] dip, MMMC[2] spray and $Mg(HCO_3)_2$ dip.

Reading Number	Treatment		
	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
1	0.62	0.90	2.11
2	1.24	0.88	2.20
3	1.12	0.88	2.44
4	1.16	0.73	1.98
5	1.15	0.77	1.98
6	1.09	0.66	1.86
7	1.26	0.56	2.47
8	1.25	0.69	2.13
9	1.41	1.48	2.19
10	1.33	1.05	2.16
mean	1.16	0.86	2.15
S.D.	0.21	0.26	0.19

[1]Colour difference is calculated using the mean colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oo solution #2 whereas the spraying solution is Wei T'oo solution #12.

Table V.2: Colour difference readings[1] (CIELAB units) of naturally aged cellulosic textiles upon treatment with the the deacidifying agents, MMMC dip[2], MMMC spray[3] and $Mg(HCO_3)_2$ dip.

Sample No.[3]	Reading No.	Treatment		
		MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
1	1	1.16	1.76	0.32
	2	0.85	1.68	0.39
	3	1.02	1.48	0.42
	4	0.89	1.45	0.37
	5	1.01	1.79	0.30
	6	0.81	1.53	0.28
	mean	0.96	1.62	0.35
	S.D.	0.13	0.15	0.06
2	1	2.03	2.20	0.31
	2	2.28	1.79	0.14
	3	1.51	2.08	0.41
	4	1.54	2.00	0.67
	5	2.10	1.92	0.73
	6	2.36	2.34	0.24
	mean	1.97	2.06	0.41
	S.D.	0.36	0.20	0.24
3	1	1.86	1.90	1.13
	2	1.48	0.72	0.69
	3	2.14	1.27	0.76
	4	4.29	1.33	1.35
	5	1.37	1.64	3.15
	6	2.36	2.31	0.71
	mean	2.25	1.53	1.30
	S.D.	1.07	0.55	0.95
4	1	0.84	1.17	1.07
	2	0.63	1.58	1.42
	3	0.67	0.71	2.01
	4	0.96	2.17	1.31
	5	0.88	0.87	2.00
	6	1.08	3.60	2.39
	mean	0.84	1.68	1.70
	S.D.	0.17	1.08	0.51
9	1	4.58	4.84	0.69
	2	1.20	3.08	1.72
	3	0.70	2.83	2.79
	4	1.25	2.01	0.21
	5	2.67	2.03	0.81
	6	2.34	2.14	0.76
	mean	2.12	2.82	1.16
	S.D.	1.42	1.09	0.94
11	1	0.88	0.92	0.55
	2	0.93	0.89	0.52
	3	0.93	1.08	0.52

	4	1.20	0.82	0.44
	5	0.89	0.71	0.47
	6	1.03	0.83	0.43
	mean	0.98	0.88	0.49
	S.D.	0.12	0.12	0.05
12	1	2.58	1.79	1.38
	2	2.60	2.07	1.18
	3	3.86	2.03	1.17
	4	1.92	2.60	1.95
	5	0.79	2.43	1.01
	6	0.99	2.68	0.78
	mean	2.12	2.27	1.25
	S.D.	1.15	0.36	0.40
13	1	2.47	1.93	0.28
	2	2.54	1.92	0.94
	3	3.06	2.79	0.49
	4	3.14	2.97	1.26
	5	2.86	1.61	0.65
	6	3.32	1.61	1.50
	mean	2.89	2.14	0.85
	S.D.	0.34	0.59	0.47
16	1	2.02	1.21	0.76
	2	2.16	2.34	0.59
	3	1.98	1.97	0.54
	4	1.94	2.53	0.86
	5	1.45	2.59	0.58
	6	1.58	1.69	0.77
	mean	1.85	2.06	0.68
	S.D.	0.28	0.54	0.13
17	1	2.69	2.37	0.25
	2	2.62	2.19	0.26
	3	2.29	2.02	0.67
	4	2.60	2.00	0.73
	5	2.35	2.24	0.83
	6	2.80	2.07	0.34
	mean	2.56	2.15	0.51
	S.D.	0.20	0.14	0.26

[1]Colour difference is calculated using the mean colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oe solution #2 whereas the spraying solution is Wei T'oe solution #12.
[3]Refers to the sample numbers as indicated in Appendix I.

Table V.3: Colour difference readings (CIELAB units) [1] of cotton textiles, aged by accelerated means, after treatment with the deacidifying agents, MMMC[2] dip, MMMC[2] spray and $Mg(HCO_3)_2$ dip.

Sample Description	Reading Number	Treatment		
		MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
irradiated -50 Mrad	1	4.80	4.80	4.72
	2	5.33	6.61	10.76
	3	5.88	4.80	6.61
	4	7.07	6.42	13.26
	5	5.98	4.46	9.32
	6	5.12	4.67	5.34
	mean	5.70	5.29	8.29
	S.D.	0.81	0.96	3.31
irradiated -75 Mrad	1	9.00	4.99	7.44
	2	6.98	5.90	9.35
	3	5.40	5.72	8.19
	4	5.81	6.33	9.11
	5	5.50	6.35	11.33
	6	5.85	6.59	7.66
	mean	6.42	5.98	8.84
	S.D.	1.38	0.58	1.44

[1]Colour difference is calculated using the mean colour of the water-treated control fabric as the initial value.
[2]MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oo solution #2 whereas the spraying solution is Wei T'oo solution #12.

Table V.4: Colour difference readings (CIELAB units)[1] of commercially dyed cotton textiles upon treatment with the deacidifying agents, MMMC dip[2], MMMC spray[3] and $Mg(HCO_3)_2$ dip.

Colour	Reading Number	Treatment		
		MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
yellow	1	2.22	1.87	1.08
	2	2.41	2.76	1.09
	3	2.23	2.59	1.07
	4	2.24	2.64	1.41
	5	2.36	2.59	0.92
	6	2.30	2.28	1.04
	7	2.52	2.45	0.95
	8	2.28	2.18	1.17
	9	2.07	2.24	1.08
	10	2.23	1.87	0.83
	mean	2.29	2.35	1.06
	S.D.	0.12	0.31	0.16
green	1	0.42	0.84	0.91
	2	0.31	0.54	0.84
	3	0.73	0.81	0.76
	4	0.84	0.50	1.20
	5	0.68	0.87	1.37
	6	1.09	0.52	0.62
	7	1.04	0.58	1.37
	8	0.52	0.75	1.40
	9	0.77	0.84	0.43
	10	0.60	0.70	0.67
	mean	0.70	0.70	0.96
	S.D.	0.25	0.15	0.35
mauve	1	0.66	0.51	0.69
	2	0.71	0.72	0.76
	3	1.45	0.81	0.63
	4	1.21	0.88	0.76
	5	1.37	0.59	0.82
	6	0.92	0.62	0.82
	7	0.99	0.78	0.84
	8	1.48	1.33	0.96
	9	1.14	1.06	0.81
	10	1.53	0.68	1.06
	mean	1.15	0.80	0.82
	S.D.	0.32	0.24	0.12
coral	1	0.62	0.92	0.79
	2	0.57	0.73	0.81
	3	1.00	0.65	0.76
	4	0.65	0.68	0.58
	5	0.62	0.78	1.39
	6	0.98	0.57	1.14
	7	0.85	0.73	0.71
	8	0.91	0.79	1.05
	9	1.29	0.69	0.72

	10	0.81	0.62	1.18
	mean	0.83	0.72	0.91
	S.D.	0.23	0.10	0.26
navy blue	1	0.83	0.67	0.65
	2	0.65	0.44	0.35
	3	0.23	0.39	0.67
	4	0.69	0.72	0.81
	5	0.85	1.03	1.35
	6	0.90	0.71	0.30
	7	0.99	0.94	0.45
	8	0.63	0.46	1.24
	9	0.76	1.22	0.61
	10	1.00	0.43	0.34
	mean	0.75	0.70	0.68
	S.D.	0.23	0.29	0.37
light blue	1	0.26	0.48	0.91
	2	0.43	0.25	0.59
	3	0.42	0.26	0.99
	4	0.64	0.27	0.85
	5	0.98	0.36	1.03
	6	0.92	0.49	0.54
	7	0.89	0.18	0.31
	8	0.86	0.35	1.17
	9	0.70	0.55	0.65
	10	0.35	0.41	0.54
	mean	0.65	0.36	0.76
	S.D.	0.26	0.12	0.27
aqua	1	1.07	0.67	0.69
	2	1.17	0.43	0.75
	3	0.98	0.76	0.76
	4	1.18	0.64	0.74
	5	1.24	0.25	0.65
	6	1.50	0.46	0.87
	7	1.18	0.76	0.67
	8	1.37	0.56	0.51
	9	0.94	0.67	0.86
	10	1.38	0.57	0.83
	mean	0.65	0.36	0.76
	S.D.	0.26	0.12	0.27

[1] Colour difference is calculated using the mean colour of the water-treated control fabric as the initial value

[2] MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oe solution #2 whereas the spraying solution is Wei T'oe solution #12.

Table V.5.1: Warp and weft bending length (c)[1] in the warp and weft direction of new cotton textiles after treatment with distilled water and deacidifying agents, MMMC dip[2], MMMC spray and $Mg(HCO_3)_2$ dip. [3]

Sample Direction	Sample Number	Treatment			
		Control	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
warp	1	2.42	5.63	3.00	2.69
	2	2.54	4.99	3.49	2.78
	3	2.74	5.44	3.25	2.87
	4	2.59	6.38	3.52	2.75
	5	2.59	4.65	3.64	2.88
	6	2.40	4.74	3.27	2.79
	7	2.55	4.85	3.59	2.95
	8	2.53	5.25	3.88	2.77
	9	2.64	5.34	3.63	2.83
	10	2.63	5.50	3.57	2.79
	sum	25.63	52.77	34.84	28.10
	mean	2.56	5.28	3.48	2.81
weft	1	1.80	3.59	2.52	2.03
	2	1.68	3.65	2.42	1.92
	3	1.82	3.84	2.52	1.98
	4	1.78	3.63	2.18	2.05
	5	1.79	3.72	2.22	1.92
	6	1.69	3.58	2.52	1.89
	7	1.73	3.53	2.19	1.95
	8	1.78	3.77	2.23	1.83
	9	1.70	4.05	2.27	2.10
	10	1.77	3.63	2.34	1.85
	sum	17.54	36.99	23.41	19.52
	mean	1.75	3.70	2.34	1.95
	W[4] ($mg.cm^2$)	11.6247	12.1783	11.8589	12.0779

[1] Bending length (c) is 0/2, where 0 = length of the overhang, cm.
[2]MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oo solution #2 whereas the spraying solution is Wei T'oo solution #12.
[3]Average of 4 readings per sample.
[4] W = weight per unit area, mg/cm^2 used in the calculation of flexural rigidity, G, where $G = W \times c^3$, mg.cm. The overall flexural rigidity, $G_o = (G_w G_f)^{1/2}$, where G_w = warp flexural rigidity and G_f = weft flexural rigidity.

Table V.5.2: Flexural rigidity in the warp direction (G_w) or in the weft direction (G_f) of new cotton fabric after treatment with distilled water or the deacidifying agents, MMMC[1] dip, MMMC[1] spray or $Mg(HCO_3)_2$ dip[2].

Sample Direction	Sample Number	Treatment			
		Control	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
warp	1	163.732	2167.476	320.190	235.088
	2	190.495	1513.172	504.104	258.084
	3	239.129	1960.575	407.094	284.019
	4	201.968	3081.543	515.015	251.173
	5	201.968	1224.462	571.937	287.004
	6	160.700	1296.945	412.756	262.292
	7	192.754	1389.350	548.691	310.056
	8	187.140	1762.238	663.649	255.304
	9	213.891	1854.430	564.896	272.289
	10	210.266	2026.165	537.308	248.442
	mean	196.204	1827.636	504.564	266.375
	S.D.	23.241	546.330	99.185	22.160
weft	1	67.795	563.469	188.650	98.810
	2	55.120	592.195	167.030	84.816
	3	70.081	689.573	188.650	93.041
	4	65.561	580.111	122.017	104.048
	5	66.671	624.401	128.874	84.816
	6	56.110	556.435	188.650	81.543
	7	60.189	533.413	124.560	93.041
	8	65.561	649.952	130.627	73.411
	9	57.112	809.006	137.800	111.849
	10	64.428	580.111	151.947	76.469
	mean	62.863	617.867	152.881	90.184
	S.D.	5.309	81.734	28.073	12.266

[1] MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oe solution #2 whereas the spraying solution is Wei T'oe solution #12.
[2] These figures have been rounded out to whole numbers and included in Table 4.5 with their corresponding 95 % confidence intervals.

Table V.6: Moisture regain values (%) of new cotton fabric after treatment with distilled water or the deacidifying agents, MMMC dip[1], MMMC spray[1] or $Mg(HCO_3)_2$ dip.

Sample Number	Treatment			
	Control	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
1	8.39	8.68	8.61	8.45
2	8.19	8.67	8.78	8.42
3	8.15	8.54	8.62	8.38
4	8.17	8.67	8.66	8.56
5	8.15	8.54	8.47	8.31
6	8.11	8.59	8.75	8.41
7	8.22	8.74	8.60	8.44
8	8.21	8.50	8.67	8.39
9	8.17	8.72	8.74	8.40
10	8.18	8.64	8.63	8.32
mean	8.19	8.63	8.65	8.41
S.D.	0.08	0.08	0.08	0.07

[1] MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'o® solution #2 whereas the spraying solution is Wei T'o® solution #12.

Table V.7: Flex-abrasion (number of cycles to rupture) of new cotton fabrics after treatment with distilled water or the deacidifying agents, MMMC dip[1], MMMC spray[1] and $Mg(HCO_3)_2$ dip.

Sample Number	Treatment			
	Control	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
1	942	130	157	147
2	864	110	150	156
3	982	145	169	185
4	809	132	152	129
5	839	149	189	149
6	872	144	136	186
7	840	138	182	163
8	858	145	152	153
9	930	155	153	158
10	975	132	152	157
11	946	149	169	150
12	990	142	162	131
13	1004	157	176	153
14	852	142	160	144
15	942	140	160	180
16	853	142	171	134
17	840	171	149	134
18	879	150	154	170
19	793	135	143	172
20	818	150	171	125
mean	891	143	160	154
S.D.	66	12	13	18

[1]MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'o® solution #2 whereas the spraying solution is Wei T'o® solution #12.

Table V.8: Flex abrasion (number of cycles to rupture) of naturally aged cellulosic textiles after treatment with distilled water or the deacidifying agents, MMMC dip[1], MMMC spray[1] and $Mg(HCO_3)_2$ dip.

Aged Sample Number[2]	Reading Number	Treatment			
		Control	MMMC dip	MMMC spray	$Mg(HCO_3)_2$ dip
3	1	19	15	8	16
	2	16	13	10	9
	3	17	12	9	8
	4	15	13	10	10
	5	22	18	7	11
	6	16	14	7	13
	7	21	16	8	15
	8	12	19	5	16
	9	17	16	9	10
	10	18	20	10	8
	11	20	12	13	16
	12	19	20	13	7
	13	20	20	16	11
	14	16	19	11	10
	15	15	17	10	11
	16	10	16	9	9
	17	17	14	8	9
	18	19	18	9	10
	19	15	16	10	11
	20	17	16	7	11
	mean	17	16	9	11
	S.D.	3	3	2	3
13	1	17	12	12	22
	2	37	15	13	14
	3	25	32	11	27
	4	26	21	16	20
	5	20	26	21	16
	6	30	15	21	16
	7	19	17	16	16
	8	14	19	10	15
	9	31	20	14	19
	10	28	23	17	11
	11	14	15	7	19
	12	23	16	10	24
	13	20	15	23	17
	14	14	12	16	18
	15	17	15	13	12
	16	23	12	22	20
	17	22	18	16	17
	18	23	15	12	19
	19	25	18	14	13
	20	21	19	23	20
	mean	22	18	15	18
	S.D.	6	5	5	4
16	1	68	83	101	67
	2	91	76	85	11
	3	101	120	8[illegible]	30
	4	103	74	96	73
	5	23	51	103	57
	6	57	86	93	70
	7	115	76	98	75

	8	118	97	81	48
	9	82	89	100	58
	10	15	63	77	76
	11	93	64	19	51
	12	97	43	97	31
	13	29	42	24	48
	14	35	63	91	34
	15	43	56	89	65
	16	101	80	112	27
	17	52	18	44	53
	18	68	85	95	85
	19	132	98	42	48
	20	51	87	92	88
	mean	74	73	81	55
	S.D.	34	23	27	21
17	1	17	17	19	29
	2	16	19	10	24
	3	32	32	10	10
	4	9	14	21	11
	5	7	13	29	31
	6	14	35	50	10
	7	57	26	42	24
	8	19	69	48	21
	9	13	68	11	11
	10	57	34	11	41
	11	46	41	17	12
	12	15	31	24	14
	13	10	48	35	23
	14	45	65	36	13
	15	25	47	51	17
	16	45	21	9	35
	17	15	31	47	24
	18	11	21	17	22
	19	61	19	12	45
	20	19	18	66	47
	mean	27	33	28	23
	S.D.	18	18	17	12

[1] MMMC is methoxy magnesium methyl carbonate. The dipping solution is the commercially available Wei T'oe solution #2 whereas the spraying solution is Wei T'oe solution #12.
[2] The aged sample number corresponds to the description found in Appendix 1.